Corrosion

IN THIS ISSUE:

FOCUS ON CATHODIC PROTECTION

Value of Corrosion Protection on Pipe Line Systems ... 9

Cathodic Protection of Large Water Storage Tanks ... 12

Methods of Cathodic Protection Coordination ... 20

Cathodic Protection of Lacq Field Well Casings ... 14

Pipe-Type Cables Protected by Shunt Resistor System ... 24

Sunspot Activity Correlates With Pipeline Voltage ... 28

Materials Problems Associated With Uncooled Rocket Nozzles 77

Performance of Zinc Anodes 81

Oxidation and Corrosion of Zirconium and Its Alloys 86

High Temperature Lubrication in Reactive Atmospheres 101

Leaching Action of High Purity Water on Materials 113

Coating Performance in Marine Environment Evaluated 117

Also: Record and Report Section, with new commercial and technical developments

Complete Contents on Pages 4 and 5

CORROSION CONTROL
PROTECTS INDUSTRY
NATIONAL ASSOCIATION OF CORROSION ENGINEERS

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OF
CORROSION ENGINEERS

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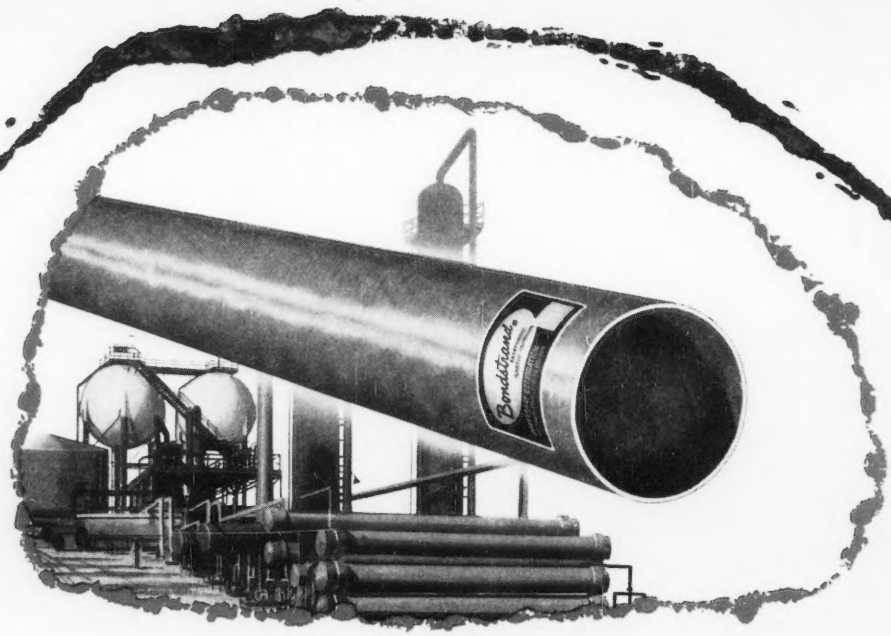

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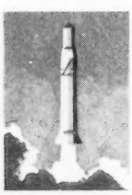

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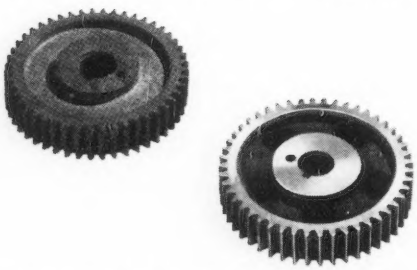

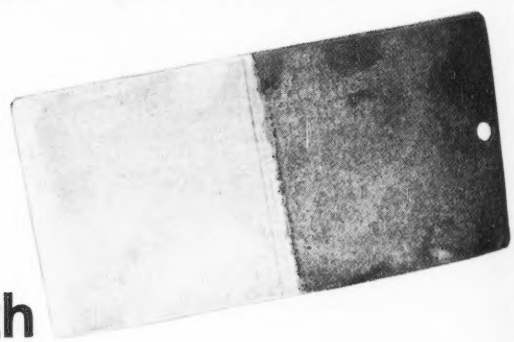

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This Month in Corrosion Control . . .

APPRECIATION OF THE WORK of Corrosion engineers in controlling corrosion of underground pipelines by a man who was there in the beginning is expressed in an article beginning on Page 9. Savings in money and good will are acknowledged by this writer, who heads one of the nation's largest pipeline systems.

10-YEAR RECORDS on cathodic protection systems are not common. A report on a decade of experience with large water storage tanks will be found on Page 12.

OILWELL CASINGS in the Lacq field in southwestern France are cathodically protected against external corrosion, especially stray currents from a nearby electrified railway. A thorough description of the methods used and an interesting discussion of the influence of geologic formations begins on Page 14.

INTERFERENCE BETWEEN CATHODIC protection systems sometimes assumes much the same proportions as stray current problems did during the heyday of electrified street railways. How a California utility coordinates its systems with others according to the recommendations of the NACE Correlating Committee on Cathodic protection is revealed in an article beginning on Page 20.

HOW A SHUNT SYSTEM works in protecting the 110-KV underground power cables and lead sheathed cables of a California utility is told beginning on Page 24. Useful design and measurement techniques are described.

SUNSPOTS INFLUENCE drastically currents of large magnitude and voltages sometimes encountered on underground pipelines. A record of how the sun's activity affects currents on a 24-inch products line is shown on Page 28.

ZINC DUST PAINTS proved the best coating for navigational buoys in four-year tests in German coastal waters. This information will be found in a news item on Page 32 concerning the Sixth International Technical Conference on Lighthouse and Other Aids to Navigation to be held next month.

MORE HELP FOR MEN who need some formal instruction in corrosion control. On Page 47 information is given on five corrosion control short courses in which NACE is participating.

IMPORTANT DESIGN information, factors of water aggressiveness and resistivity, anode materials, rectifier settings and other information useful in protecting potable water tanks cathodically is contained in an article beginning on Page 96.

IMPORTANCE OF SMALL percentages of certain metals in zinc anodes used as a source of cathodic protection energy in sea water is revealed in an article beginning on Page 81. Controlled tests over a 36-week period are described in detail.

ROCKET MATERIALS' resistance to the corrosive effects of hot gases and oxygen at high velocities are explored in two articles. One, concerning rocket nozzles, begins on Page 77 and the other, concerning ablation behavior begins on Page 91. Use of the discharge from a 1000 kw plasma arc in the ablation tests is described.

ZIRCONIUM CONTINUES to get studied attention from engineers concerned with finding materials qualified to give the reliability required of atomic energy systems. Arc-melted zirconium's reaction to 300 C high purity water is described on Page 86.

CONTROLLING CHEMICAL ACTIVITY of the surfaces of bearings is essential if corrosion is not to take place. Beginning on Page 101, the problem of lubrication at high temperatures in reactive atmospheres is weighed. Gaseous halogen-subsituted methane derivatives have promise.

EXTREMELY SENSITIVE reactions on thin-film corrosion indicators permit anticipating corrosion, something which has not been possible heretofore with other systems. This method, which assesses the electrical resistance of metallic films vacuum-deposited on non-metallic surfaces, permits remote monitoring of materials isolated from environments. Visual indicators also are available. See Page 105.

ORGANICS ARE LEACHED from plastics by ultra-high purity water at rates which often makes these materials unsuitable for equipment handling it. Data on extensive tests covering natural and synthetic rubber, polyethylene, two epoxy resins, and two phenol-formaldehyde resins are given beginning on Page 113.

STATISTICAL ASSESSMENT of coatings performance sometimes is difficult. One means of doing it is given in an article beginning on Page 117.

SOME INTERESTING CONCLUSIONS about the mechanism of aerobic corrosion of 1036 steel are reached in an article beginning on Page 121. Influence of oxygen concentration cells on the steel when exposed to brine is considered. Tabulated data relating pH to fatigue life are given.

CONTENTS

Page

This Month in Corrosion Control 3

Beware of Ground Return Circuits in Electrification—An Editorial 7

TECHNICAL TOPICS

Page

Management's Viewpoint: Value of Corrosion Protection on Pipe Line Systems
By Harry K. Phipps 9

10 Years of Cathodic Protection for Large Water Storage Tanks
By H. M. Patton, Jr. 12

Engineering and Design Problems Affecting Cathodic Protection of Lacq Field Well Casings
By M. Estavoyer 14

Other Papers on Cathodic Protection Listed 18

Suggested Methods and Practices for Cathodic Protection Coordination
By David T. Jones 20

Novel Distributed Cathode Systems Are Used on Telephone Cables
By Douglas T. Rosselle 22

Pipe-Type Cables Protected by Shunt Resistor System
By H. H. deLaneux and H. A. Medlock 24

Sunspot Activity Correlates With Pipeline Voltage 28

RECORD AND REPORT

Page

Soybean Processing Plant Stopped by Corrosion 29
Two Steels Are Used to Solve Auto Oil Filter Corrosion 29
Technical Writing Course 29
Niobium Production To Be Achieved by New Process 29
Book News 29
Free Literature List Available Covering 1945-1960 30
New Device Inspects Equipment in Use By Ultrasonic Pulse 32
Plastics Conference Dates 32
Unusual Model Helps Study of Electronic Properties of Metals 32
Buoys Protected by Zinc-Dust Paints on Sandblasted Surfaces 32
Electric Motor Components Coated for Corrosive Exposure Service 34
Welding Society to Meet 34
Water Pollution Conference 34
ASEE Elects New Officers 34

Page

NBS Conducts Study to Eliminate Rapid Oxidation of Niobium 34
Construction Begins on New Laboratory Site for NBS 34
German Standards 34
Environmental Terminology 34
French Monorail Demonstration Indicates Advantages of System 38
High Strength Alloy Obtained by Adding Fibers to Metals 38
Electrochemical Meeting to Include Corrosion Sessions 38
Navy Builds Laboratory for Measuring Magnetic Fields 38
Cable Withstands 20-Year River Exposure 40
First of Spectral Data Sheets Published 40
New Products 43
Men in the News 46
Technical Articles Scheduled for September 127

Index to Advertisers 6

Advertised in This Issue 6

Corrosion Abstracts 65

NACE NEWS

Page

Program Information Given on Five Corrosion Short Courses 47
Northeast Region News
Time Schedule and Abstracts Given for Oct. 11-14 Huntington Conference 48
Center of Heavy Industrial Region To Be Site of 1961 Corrosion Show 51
Section Calendar 51
1960 Corrosion Show Described in Color Slide Presentation 51
South Central Region News
Seven Abstracts Given From Tulsa Program 51
Canadian Region News
Edmonton Section 51
Western Region News
Nuclear and Missile Symposium To Be Included at San Francisco Conference 52
San Joaquin Corrosion Tour To Be Held October 12-13 53
Portland Section 53
Central Arizona Section 53
National and Regional Meetings and Short Courses 55
Free Radicals May Be New Source of Useful Energy 55
Alternate Offices Built in Case of Nuclear Attack 55
Corrosion Magazine Binders Available from NACE Office 55
Alloy Casting Literature 55
Annual Titanium Conference 56
South Central Region News
Alamo Section 56
Permian Basin Section 56
Region Plans Exhibition for 1961 Houston Meeting 56
North Central Region News
Corrosion Problems of Four Industries To Be Discussed at Milwaukee Meeting 58
Twin Cities Section 59
Southeast Region News
Ohio Valley Section 60
Foreign Corrosion Reports 60

DIRECTORIES

Page

NACE Officers, Directors and Staff Members 76
NACE Region and Section Officers 125

CONTENTS

TECHNICAL SECTION

Page

Materials Problems Associated With Uncooled Rocket Nozzles
By Jesse D. Walton, Jr. and C. R. Mason, Jr. 77

Effect of Iron, Aluminum and Cadmium Additions on the Performance Of Zinc Anodes in Sea Water
By L. J. Waldron and M. H. Peterson 81
Discussion: J. A. H. Carson, Pages 84-85

Oxidation and Corrosion of Zirconium and Its Alloys (Oxide Film Breakdown in Arc-Melted Sponge Zirconium)
By B. Cox 86

Ablation Behavior of Materials Subjected to Missile Re-Entry Heat Flux Rates
By R. M. Krupka and D. E. Taylor 91

Cathodic Protection for Metallic Water Tanks
By Conrad A. Bauer and Levi C. Eddy 96

High Temperature Lubrication in Reactive Atmospheres
By R. L. Johnson, M. A. Swikert and D. H. Buckley 101
Discussion: Roy E. Dial; Page 104; Reply by Robert L. Johnson, Page 104

Development of Thin Metal Film Corrosion Indicators
By David Roller and G. H. Rohrback 105

A Chromatographic Method for the Separation Of Saturated Hydrocarbons from Creosote
By Richard W. Drisko and Harry Hochman 111

Leaching Action of High Purity Water On Plastics and Other Materials
By E. L. Knoedler and J. B. Goodson 113

Coating Performance in a Marine Environment Evaluated by Statistical Methods
By Frank E. Cook and Arthur S. Marthens 117

Effect of pH in the Range 6.6-14.0+ On the Aerobic Corrosion Fatigue of Steel
By F. J. Radd, L. H. Crowder and L. H. Wolfe 121
Discussion: A. V. Alessandria, F. Lee Current, Page 124; Replies by F. J. Radd, Page 124

Technical Articles Scheduled for September

Problems Encountered in Cathodically Protecting Pipelines Crossing Railroads, by F. L. Chatten

Inhibition of Corrosion of the Hypereutectic Aluminum-Silicon Automobile Engine, by H. Lee Craig and P. H. Woods

Compositional Effects in Corrosion of Types 347 and 316 Stainless Steel in Chemical Environments, by C. P. Dillon

A Chromatographic Method for Separation of Saturated Hydrocarbons From Creosote, by R. W. Drisko and Harry Hochman

Soil Microorganisms in Relation to Cathodic Protection, by J. O. Harris

Hydrogen Permeation Through Metals, Alloys and Oxides at Elevated Temperatures, by C. L. Huffine and J. M. Williams

Laboratory Investigation of Water Side Scale and Corrosion in Presence of High Process Side Temperature, by Herman Kerst

Concentration Cells and Aqueous Corrosion, by Edward Schaschl and G. A. Marsh

Selective Removal of Chromium From Type 304 Stainless Steel by Air Contaminated Lithium, by Robert E. Seebold, L. S. Birks and E. J. Brooks

Qualitative Mechanism of Stress Corrosion Cracking of Austenitic Stainless Steels, by D. van Rooyen

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ADVERTISED IN THIS ISSUE

A

ANODES
- Galvanic 13
- Graphite 11
- Magnesium 13, 16, 18, 25
- Plating 13

B

Backfill, coke breeze..... 18

C

CATHODIC PROTECTION
- Engineering ...16, 18, 19, 21, 23
- Flange tester 6
- Probe bar 6
- Rectifiers 8, 10, 18, 24
- Voltmeter, pipe-to-soil 6
- Weld connections 17

COATINGS
- Chlorinated rubber 63
- Coal tar 26, 31
- Coal-tar epoxy 28
- Cold applied 44, 64
- Hot applied 44
- Industrial 33, 54
- Maintenance 57
- Mastic 35
- Metallic 59
- Primers 26, 57
- Services 33, 59
- Synthetic rubber 69
- Pipeline 15, 35, 42
- Corrosion Engineering..... 19

F

Fiberglass 52

G

Galvanizing materials 13
Galvanizing, hot dip 59

I

INHIBITORS
- Diamine 49
- Iron and steel parts 2
- Petroleum 1
- Pickling acids 36, 37

INSTRUMENTS
- Flange tester 6
- Voltmeter, pipe-to-soil 6

L

Lead 13

P

PIPE
- Asbestos cement 66
- Asbestos felt 28
- Felt 44
- Glass wrap 44
- Plastic 45, 61
- Plastic lined 23
- Polyethylene 27
- Seals 28, 40
- Wrapping 28, 59
- Coatings 26

PLASTIC
- Ducts and stacks 52
- Fittings 61
- Laminates 45
- Pipe 45, 61
- Pumps 61
- Valves 61

Plating materials 13
Pickling services 59

R

Rectifiers 8, 10, 24

S

Salt, Sea synthetic 46
Sandblasting 59
Stainless steel 61
Surveys 18, 19

T

TAPES
- Coal tar 67
- Polyethylene, pressure sensitive 59

Testing laboratories 19

V

Valves 58, 61

W

Water treatment engineering.. 19
Welded connections 17
Wrought iron 50

INDEX TO ADVERTISERS

Vol. 16 August, 1960 No. 8

- Agra Engineering Co. 6
- Allied Chemical Corporation Semet-Solvay Petrochemical Div. 27
- Alloy Steel Products Company, Inc. 58
- Amchem Products, Inc. 36, 37
- Amercoat Corporation .....Inside Front Cover
- American Cyanamid Plastics and Resins Div. 45
- Burndy Corporation 17
- Byers, A. M. Company Wrought Iron 50
- Carboline Company 57
- Cathodic Protection Service 21
- Chase & Sons, Inc. 39
- Corrosion Engineering Directory 19
- Corrosion Rectifying Company 18
- Corrosion Services, Inc. 16
- Culbertson Company, Syd E., Inc. 15
- Day, S. D. Company 44
- Dowell 1
- du Pont, E. I. de Nemours & Co., Inc. 69
- Duriron Company, The .....Inside Back Cover
- du Verre, Incorporated 52
- Eicor, A Division of The Scranton Corporation 24
- Federated Metals Division American Smelting & Refining Company. 13
- Good-All Electric Manufacturing Co. 10
- Harco Corporation, The 23
- Hagan Chemicals & Controls, Inc. 75
- Hercules Powder Company Cellulose Products Department 63
- Humble Oil & Refining Company 42
- International Nickel Company, Inc. Primary Nickel Department 41
- Johns-Manville Corporation Industrial Water Pipe 66
- Koppers Company, Inc. Tar Products Division 31
- Lake Products Company 46
- Midwestern Pipe Line Products Company.. 56
- Nalco Chemical Company 49
- Napko Corporation Industrial Coatings Division 33
- National Association of Corrosion Engineers 30, 55, 62, 72, 73, 74, 128
- National Carbon Company, Division of Union Carbide Corporation 11
- Perrault Supply Company Div. of Allied Paint Mfg. Co. 28
- Pittsburgh Chemical Company Protective Coatings Division .....Back Cover
- Positions Wanted and Available 48
- Price, H. C. Company Pipe Coating Division 35
- Radio Receptor Company Division of General Instrument Corp. 8
- Reilly Tar & Chemical Corp. 26
- Royston Laboratories, Inc. 64
- Saran Lined Pipe Company 61
- Smith, Nowery J. Company 59
- Solvay Process Division Allied Chemical Corporation 2
- Standard Magnesium Corporation 25
- Standard Pipeprotection, Inc. 53
- Tapecoat Company, The 67
- Tube-Kote, Inc. 54
- Wholesale Coke Company 18
- Williamson, T. D., Inc. 40

Beware of Ground Return Circuits in Electrification

MUCH TO THE DISMAY of many corrosion engineers, a scheme has been proposed whereby electric power will be brought to rural areas in India using earth for return. This is especially significant because coincident with the demise of most of the electrified street railways in this country, the problems of stray current corrosion caused by ground return from this source have almost reached the vanishing point. This new scheme may reproduce in India a situation which caused untold millions in damage to underground piping and cables and caused the expenditure of millions in control measures in the United States.

It is even more dismaying to learn that similar systems are now in use in New Zealand, Australia and Saskatchewan.* While it is true now that these systems are in remote regions where underground metallic plant is limited, it does not follow that this always will be the case. Initially the poor grounding of electrified railways caused little damage in this country, but the ultimate consequences of the defective systems are too well known to merit repetition. It can be assumed that the industrialization of all nations will continue at an accelerated pace and that what may now be a remote outpost can become a thriving industrial community in 20 years or less.

A case in point may be found in this issue of CORROSION** where it is reported that the problem of preventing the corrosion of oil well casings is significantly complicated by stray current from a nearby electrified railway. In the vicinity of even the well-maintained electrified railways in this country, stray current corrosion problems are substantial; even the best engineered control measures are not wholly successful.

A corollary problem is that created by ground currents returning to high-wattage radio and television broadcasting stations. The federal government, among others, is interested in corrosion problems created in the vicinity of these powerful stations by the currents returning to ground. It would not be extraordinary to discover that these currents are behaving much the same as the ground return currents from electrified railways. The situation is not identical, however, because of the high voltages involved, so this potential source of corrosion damage may be one that needs some expert attention.

In the case of ground return rural electrification schemes—as a minimum—it is hoped some notice will be taken of the corrosion hazards involved in the system. If any incentive is needed to justify this consideration, the history of stray current corrosion in the United States and other industrialized nations should be sufficient. At the very least, in case economic and other considerations are such that the single wire system is the only feasible one, plans should be made to ultimately modernize the circuits to provide for return by metal conductor.

There will be a strong inclination to use ground return systems in countries such as India, where capital frequently is limited, because a single phase system may cost as little as 25-30 percent* of an equivalent three-phase system. Planners who have the long-term best interests of their countries at heart will want to include provisions for bringing single phase systems to standard. This might be done much in the same way that the railways, after starting with a single track, have gradually provided double track for those sections of their routes where the traffic is heaviest. Another corollary is the by-pass system used by the large trunk pipelines to increase the capacities of their lines.

---

* R. W. Atkinson and B. W. Garg, Bihar Institute of Technology, P. O. Sindri Inst., Dhanbad (Bihar) India, at American Institute of Electrical Engineers' June 23, 1960, Atlantic City, N. J.

** Engineering and Design Problems Affecting Cathodic Protection of Lacq Field Well Casings by M. Estavoyer.

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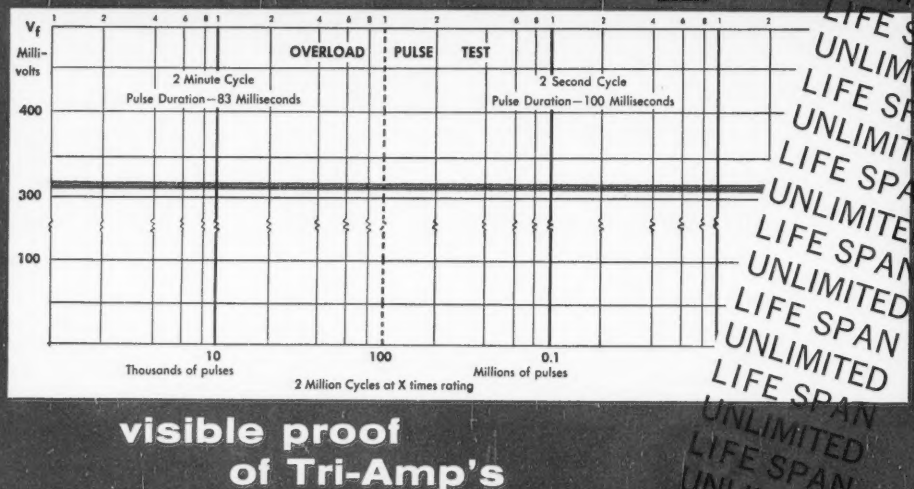

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Management's Viewpoint

Value of Corrosion Protection On Pipe Line Systems*

Harry K. Phipps
Mobil Oil Company
Wichita, Kansas

AN INVITATION to present a paper on benefits to be derived from corrosion protection of pipelines was promptly accepted because everyone responsible for the operation of any portion of our country's vast network of pipelines owes a debt of gratitude to the corrosion engineer for making his job easier.

It must be admitted, however, that the corrosion engineer has not always been held in such high esteem. In fact it is not too difficult to remember, when pipe line transportation was in its infancy, that the average pipeliner was inclined to look with some suspicion on any technical man. Those were the days when a crew made up of strong-backed men equipped with pipe tongs, a "lazy board," a collar pounder, along with the ever-present mule skinner and his wagon, was felt to be about all that was needed to build any pipe line. Right-of-way easements sometimes were obtained in a haphazard manner. Survey crews to stake the route of the line were used only on special occasions and the idea of a corrosion study was something that wasn't even remotely considered.

As already indicated, pipe line management, generally speaking, is now so well sold on the merits of sound corrosion prevention measures, that most are glad to have the opportunity to express their appreciation for the fine job being done. There are, however, so many angles by which the subject can be approached that to cover all of them would result in an article of much greater length than could be justified here. For this reason, time will be taken only to present a few arguments as to why it is definitely in management's best interest to actively support a well balanced corrosion program.

Effects of a Leak Considered

As a starting point consider the matter of downtime occasioned by a corrosion leak on a pipe line. Naturally, the over-all cost of such downtime will vary almost in direct proportion to the size of the line in question and the extent to which it is being used. In other words, time lost on a system operating at 95 percent to 100 percent of capacity will naturally be much more critical than would be in the case with one which needs to operate only 60 percent of the time in order to handle its scheduled throughput.

The area in which a leak occurs also can make for a wide variation in ultimate costs, including damage to third parties. As an illustration of this point, a line break in a pasture area remote from any stream or other body of water probably would only result in a rather modest outlay for property damage. On the other hand, a break of the same size in a congested area or immediately adjacent to a community water supply, could be exceedingly costly. Furthermore, if loss of life happened to be involved, the line might even be declared a nuisance and as a result enjoined from further operation until a thorough investigation could be made and the necessary corrective measures taken.

In addition to these more or less tangible penalties, there are still other considerations. For example, any downtime on an important section of a crude trunk line would for the time being stop all revenue for the entire line, and what is equally important, all or part of the oil supply of one or more refineries might be cut off during the shut-down period.

Over and above monetary loss, the damage to public relations which can be occasioned by a bad leak could be quite disastrous to company standing in a given community. Even though it is readily apparent that it would be quite difficult to put a price tag on such damage, nevertheless everyone has seen enough newspaper accounts of bad oil spills to know that the public reaction is usually not favorable to the company involved.

As a second step in the consideration of the topic it might be well to review briefly some of the ground that has been covered during the past 40 to 50 years with respect to the prevention of pipe line corrosion. In doing so, no attempt will be made to weigh the technical aspects of the corrosion problem.

Corrosion Damage Not Predictable

In the early days of pipe line operation, the vast majority of lines—oil, gas, or water—were laid bare. In the case of low pressure lines where it was feasible to use cast iron pipe, the corrosion problem often was negligible, but on uncoated steel pipe, the situation was something entirely different. Probably a much more accurate way of stating the matter would be to say that the amount of external corrosion that might be experienced on a given line was entirely unpredictable. As support for this statement, and as any experienced pipeliner can readily testify, when a new line was built through a new area there was no way of knowing ahead of time just what the useful life of the pipe was likely to be. In dry sandy soil life might be as long as thirty or forty years without a leak—that is, if no electrolytic condition arose to complicate the problem. On the other hand, in clay, marshy, or alkaline soils, this same bare pipe might not last six months, regardless of its original wall thickness. Nevertheless in spite of the fact that thickness alone could not be relied upon to prevent losses due to corrosion, heavy pipe continued to be specified for many years.

As many can recall, early pipe coatings, even when applied on the entire length of the line—and this was certainly the exception rather than the rule—probably did almost as much harm as good. The reason for this was that these first coatings were little more than a black paint applied cold over a given section of line. No wrappings of any kind were employed and, of course, holiday detectors were then unknown. Although these early coatings seemed to stand up well through sandy or loam soils it is likely that in this type of soil bare pipe no doubt would have done equally well.

Clay Soil Was Troublesome

It was in clay or gumbo soil, however, that these old cold coatings really gave trouble. During the rainy periods, the clay would shrink and in the process would pull off a section of the coating. Naturally, wherever the coating was broken, current discharge was accelerated with the result that penetration of the pipe wall within a matter of months was not an unusual occurence. The advent of more dependable coatings and approved methods of application, together with cathodic protection have largely solved the problem of external corrosion and with it the resultant high cost of downtime from this source.

Substantial Savings Are Made

As already indicated, the dollar and cents savings that have accrued to the average pipe line through a substantial reduction in leak frequency and the attendant minimizing of downtime is difficult to properly evaluate. Beyond a doubt, however, these savings particularly with respect to the many large diameter lines now in service have been most substantial. The truth of this

(Continued on Page 10)

★A paper presented under the title "Management's Viewpoint of Corrosion Protection of Pipe Line Systems," at a meeting of South Central Region, National Association of Corrosion Engineers, Denver, Col., October 14, 1959.

Management's Viewpoint—

(Continued From Page 9)

statement becomes even more apparent when the difference between incremental pumping costs and full tariff receipts for any throughput volume lost as the result of downtime due to leaks are considered. Then too, and as previously mentioned, this penalty is still further aggravated on lines that are operating at or near capacity.

Internal corrosion did not become a major problem until product pipe lines began coming into general use about 30 to 35 years ago. While it is true that more or less trouble of this nature has always been experienced on crude lines where oil with a high sulfur content was being transported, these instances were fortunately often isolated. When, however, gasoline and other refined products began to be moved across country at high pressures, internal corrosion became significant. Although these same products had been pumped around refineries for years, often in a sour and untreated state while usually carrying quantities of entrained moisture there was little or no pitting due to corrosion. On the first long distance product lines, however, the combination of moisture and high pressure caused pitting of inside pipe walls, which in many instances was sufficiently severe as to result in complete penetration. While the pitting itself was bad enough, the resulting accumulation of scale and other oxidized material was at times sufficiently great as to seriously curtail the capacity of a section of line. In addition, the scale caused considerable damage to pumps, strainers, valves, etc. As is now well known, the running of scrapers on a regular schedule solved the problem of scale accumulation. The pitting now is kept under control or eliminated altogether either through the use of various inhibitors or by product dehydration.

Tankage Also Is Protected

It is not only on the pipe line itself that the corrosion engineer has made such an outstanding contribution. He has been equally effective in adding to the useful life of the existing tankage at pump stations and terminals. As is well known by the members of the National Association of Corrosion Engineers, the preventive measures employed include rectifier units, magnesium anodes and the application of various types of protective coatings to the interiors of tank shells. In addition, the engineer usually sees to it that all new tank grades are designed so that they will drain properly and that these same grades are covered with sand, crushed limestone, or other material highly resistant to the flow of electric current.

Abstract

The principal reasons for adequate corrosion protection on pipelines are uninterrupted and efficient operation, preservation of good public relations, avoidance of damage claims from loss of life, fire or property damage, reduction of damage to equipment from corrosion products and full return from money invested in facilities.

History of corrosion protection on pipelines is reviewed briefly and credit is given to the corrosion engineer for the substantial savings that have been effected through his efforts. 1.2.1

As already pointed out, the degree to which the twin problems of internal and external corrosion have been conquered on pipe lines and on tankage and other metal structures can be credited largely to the patient and untiring efforts of the corrosion engineer. It is for that reason that the vast majority of pipe line companies maintain a corrosion organization which is charged not only with the responsibility of keeping their own systems operating with a minimum amount of downtime due to leaks, but also with keeping abreast of the latest developments in corrosion mitigation. In this last connection, it is well known that NACE has made an outstanding contribution in the matter of corrosion prevention, not only on pipe lines but throughout industry in general.

Origin of NACE Related

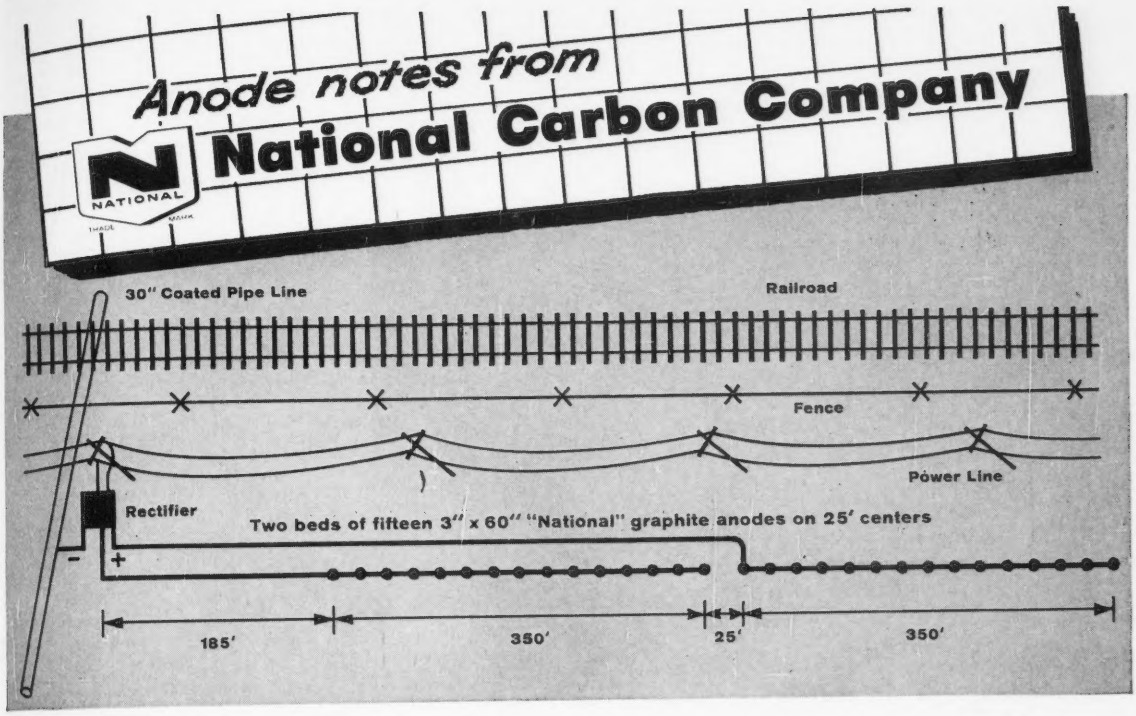
What may not be quite so well known, however, is the fact that the present NACE is the outgrowth of an organization started in March 1936 by a small group of pipe line engineers. This organization, originally known as the Mid-Continent Protection Association, remained an independent group until 1940 when it was invited to affiliate with the Petroleum Industry Electric Association. It continued as a Corrosion Section of this association until the spring of 1943 when, after being unable to get the PIEA to set up procedure that was considered essential, the group decided that a new and separate organization was necessary. An organizing committee was therefore, appointed which held several meetings during the summer of 1943, both with and without officials of PIEA. Finally in October of the same year at a meeting of eleven pipe line engineers, a preliminary draft of the first articles of organization by-laws was tentatively adopted, with the result that NACE was incorporated on October 8, 1945, as a non-profit corporation under the laws of the State of Texas.

The principal reason for this historical resume is to show that the pipe line engineer realized as early as anyone the importance of attacking the problem of corrosion in a scientific manner. For that reason he probably feels he is entitled to stick out his chest just a little whenever the over-all accomplishments of NACE are being discussed. As evidence of the support that pipe line management as well as that of industry in general has given the corrosion protection program, the vast majority of companies whose men are active in this association have taken out one or more corporate memberships.

Public Relations Value Overriding

As already emphasized, responsible pipe line management feels that any sound corrosion prevention program is fully justified from the standpoint of good public relations even if for no other reason. For example, every right of way and claims man knows that when—even for the first time—a lot of oil is spilled on a fine piece of farm land, or this same oil gets into a well or other source of water supply for a farmer's cattle, to paraphrase the late Dale Carnegie, nothing very much has been done to "make friends and influence people." This is true even when damage claims are settled on the landowner's terms. To put it still another way it is a well established fact that the majority of landowners would much prefer to avoid having a line break on their property even if the resulting settlement is entirely satisfactory from a monetary standpoint. The reason, of course, for this attitude is that almost any piece of oil-soaked ground, even under the best clean-up and rehabilitation measures, is likely to remain somewhat of an eyesore for a long time. Because, unfortunately, this sort of feeling usually prevails, even after the first oil spill, it doesn't take much imagination to see the damage that can be done to a company's public relations if leaks continue to occur at more or less regular intervals on the same piece of land. Naturally, when an oil spill occurs in a congested area where there is danger of fire, not only is there a chance of having a staggering claim for property damage, but what is much more important, there is always a possibility there may be a loss of life.

In summing up, it is clear that all farsighted pipe line management definitely views corrosion protection as something highly desirable and for that reason will lend its wholehearted support. Not only does management want to see corrosion held at a minimum to better the company's position in the eyes of the public, but also because in these days when creeping inflation is tending to push construction and maintenance costs ever higher, it is imperative that all exert the utmost effort to see that maximum return is obtained from every dollar spent. Regarding pipe lines, this means that they must be kept operating as near to 100 percent efficiency as is practical. It naturally follows that this goal is not possible if more or less regular shutdowns and delays due either to internal or external corrosion are occurring.



PROTECTION OF 645 MILES OF 30 INCH COATED STEEL PIPE LINE

In 1954 the management of a large oil company decided to protect part of their new transmission system. Since time was short they decided to contract for a turnkey cathodic protection job.

The contractor's* corrosion engineers surveyed the entire line and installed eighteen 24 volt rectifiers with 10-20 amp capacity to power the associated "National" graphite anode beds. The beds had from ten to fifty 3" x 60" graphite anodes in coke breeze backfill on 25 ft. spacing.

After the system was energized in 1955, the corrosion engineers tested and made necessary bonds with over 60 "foreign owned" pipe and cable crossings and adjusted the entire system to provide a potential of —.85 volts pipe to soil potential over its entire length.

A complete survey in 1959 showed that the anode beds are all performing satisfactorily. Several new "National" graphite anode beds will be installed to protect additions to the original pipe line. For data on graphite anodes, contact National Carbon Company, Division of Union Carbide Corporation, 270 Park Avenue, New York 17, New York. In Canada, Union Carbide Canada Limited, Toronto.

*Harco Corporation, Cleveland, Ohio

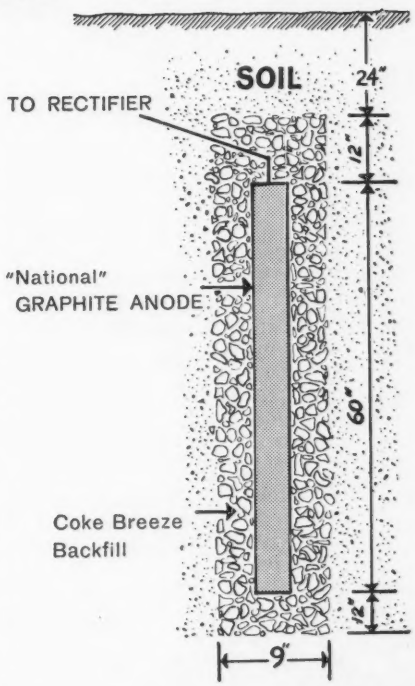


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Figure 1—Interior of a 125,000-gallon ground storage tank showing anodes suspended from the tank top. The white substance on the partially deteriorated anodes is primarily aluminum hydroxide and aluminum oxide. Part of one anode shown on the left has broken off. For overhead storage tanks, a screen guard must be used over the tank's riser to prevent fragments from anodes falling into the riser and wedging in the shut-off valve.

Abstract

Describes cathodic protection system used for 10 years successfully to control corrosion in 27 water storage tanks which are secondary water supply for company's plants. Discusses rectifiers and anodes used, advantages of cathodic system and its economics. 5.2.1

10 Years of Cathodic Protection For Large Water Storage Tanks*

H. M. Patton, Jr.

Brown Shoe Company

St. Louis, Missouri

CATHODIC PROTECTION has been used since 1944 to control corrosion in 27 water storage tanks at the Brown Shoe Company's plants. Varying in size from 40,000 to 150,000 gallons, the tanks are used as secondary water supplies for the plants' fire sprinkler systems. Most of the plants are in small communities with volunteer fire departments and limited city water supplies. These water tanks are overhead storage tanks or ground storage tanks connected to a fire pump.

An 18-volt, 10 ampere rectifier is used for each tank. Current densities vary from 0.067 milliamperes to 2.5 milliamperes per square foot of submerged tank area. The 2.5 setting for the largest tank is only 50 percent of the rectifier's maximum capacity. Exact current setting for each tank depends on the type water stored and condition of the tank shell interior.

Typical anode configuration in overhead tanks is five aluminum electrodes (½-inch in diameter by 19 feet long) suspended in a circular pattern from the tank top. The larger tanks have a 96-foot electrode suspended in the tank riser. The rectifier unit usually is mounted at the base of one tank leg and is connected to the electrodes by wires in ½-inch conduit. Holes are cut in the tank top so that electrodes can be suspended.

For ground storage tanks, the anodes extend slightly above the normal water line in the tank. The rectifier is mounted on the tank's side.

Figure 1 shows the anodes suspended in a storage tank at Union, Missouri. A white colored substance (aluminum hydroxide and aluminum oxide) has formed on the partially deteriorated anodes. This substance is porous enough to permit current passage from the anode through the oxide to the water.

The white material seen in Figure 1 on the tank sides is primarily calcium carbonates and magnesium carbonates. The water stored in the tank had considerable hardness; therefore some dissolved solids were deposited on the tank by the current passing through the water. The tank sides showed no pitting or corrosion at annual inspections made for the past 10 years. Cathodic protection has been used during this time.

Advantages of Cathodic System

Use of cathodic protection system for water tank corrosion control has two advantages over a coatings system. When a tank is coated, it is difficult to determine whether the metal is being properly protected below the coating. When cathodic protection is used, the tank surfaces can be checked carefully for pitting or other forms of corrosion.

The other advantage is that cathodic protection eliminates the need to drain the tank for a two or three day period as required if the tank is to be coated internally. Cathodic protection permits continuous water storage, an important consideration when the water is used for a plant's fire sprinkler system.

Economics of Cathodic Protection

Cathodic protection is slightly more expensive than a simple coating system to protect water storage tanks. On a 125,000 gallon ground storage tank, the initial cost of a cathodic protection system is about $700. If the tank were coated with water-proof protective materials, the cost would be about $425. The life of the coating would be about five years. On the cathodic protection system, yearly costs to maintain the system and to replace the sacrificial anodes is about $110.

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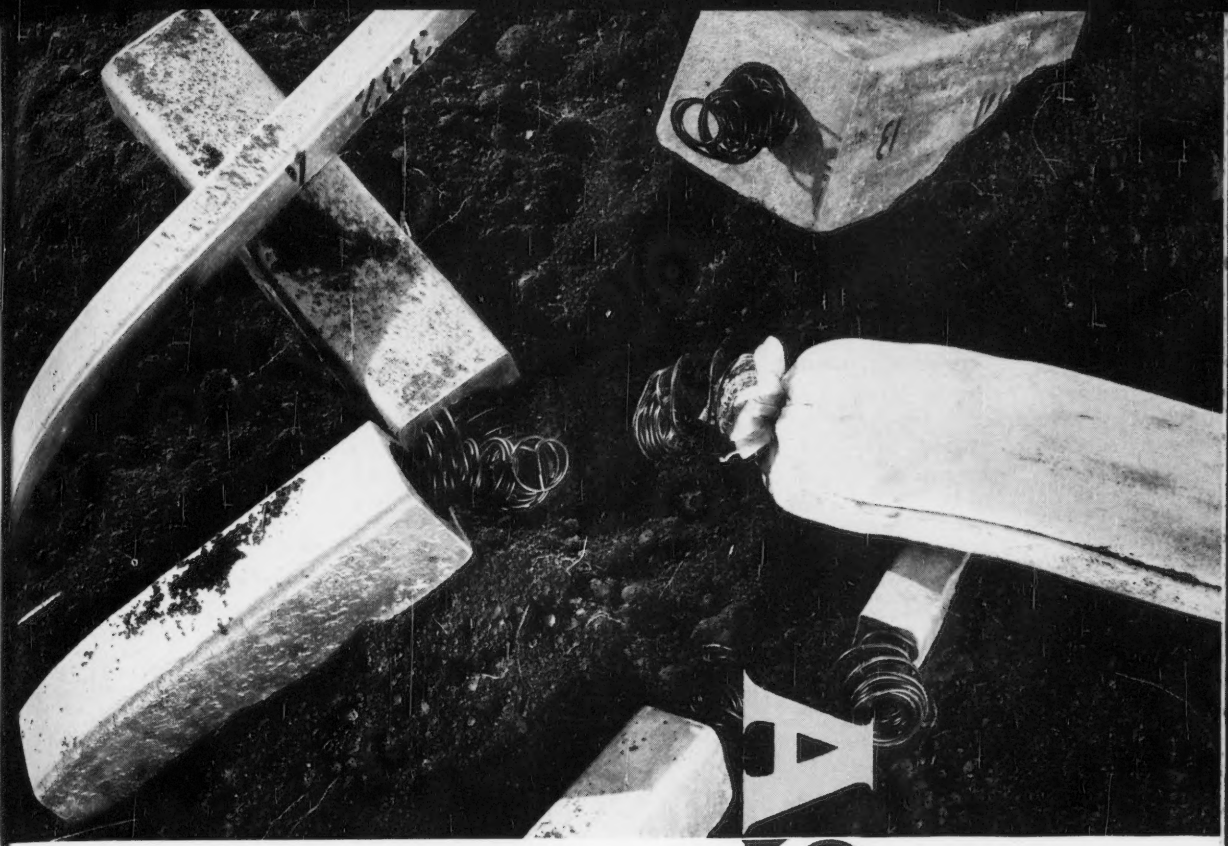
American consumers purchased five million television sets, half a million dish washers and 400 billion cigarettes last year.

★ Condensed from a paper titled "Cathodic Protection Program for Industrial Water Storage Tanks" presented at the North Central Region Conference, National Association of Corrosion Engineers, October 20-22, 1959, Cleveland, Ohio.



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Abstract

Potential profiles taken of 9⅝ inch gas well casing at the Lacq Field in southwestern France were evaluated to determine the correlation between current discharge zones and geological formations. At the same time an effort was made to distinguish between galvanic currents and stray current originating from an electric railway traversing the field. Estimates of the general electrical conformation of the field were deduced from data collected on a few wells.

Numerous conventional methods were considered in attempting to fix the optimum current density to be applied to each casing. Cathodic protection currents varied in volume from 15 to 50 amperes, so ground beds of varying configurations were used, including 50 meters of pipe horizontally in a conductive zone and 50-meter vertical holes.

An investigation is under way to determine what benefits can be obtained by altering the existing electrical configuration resulting from the electric railway stray currents. This will be accomplished by modifications at a substation and possibly by using the pipeline grid as a low-resistance drainage return. There is some concern that cathodic protection may produce local cells whose effect seems to be as severe as the galvanic currents normally existing. 5.2.4

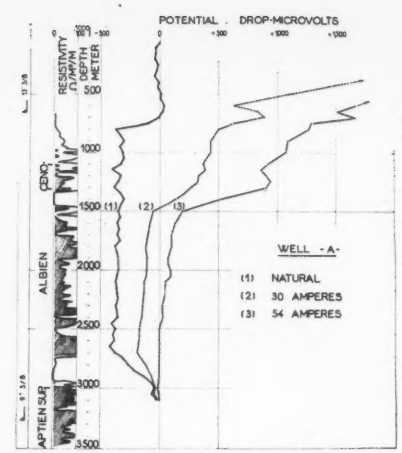


Figure 2—Potential profiles on 9⅝-inch casing in Well A.

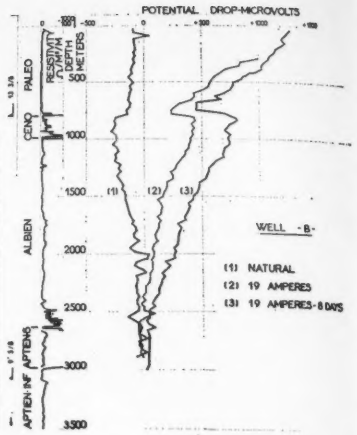


Figure 3—Potential profiles on 9⅝-inch casing in Well B.

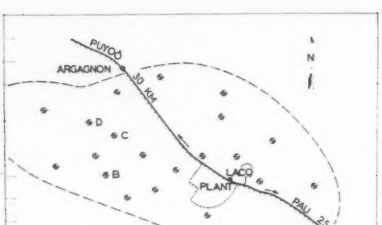


Figure 1—Map of deep Lacq Field. Letters A, B, C and D indicate some of the wells tested.

Engineering and Design Problems Affecting Cathodic Protection of Lacq Field Well Casings*

M. Estavoyer

Societe Nationale des Petroles d'Aquitaine
Paris, France

Introduction

LACQ FIELD, situated in the southwest corner of France, is a vast anticline producing sour gas containing 15 percent H_2S and 9 percent CO_2. At present the reserves are calculated to be 150 teramillion* cubic meters, representing an important source of energy for France.

The last production casing is 7 inches OD set in the top of the pay formation at depths varying from 3500 to 4600 meters. Because of the great depth and numerous difficulties encountered during drilling operations the cost of a well is extremely high.

Because in the Albian formation, a sour water zone contains NaCl and H_2S, the casing is subjected to corrosion. Moreover, a double track electric railway line having a heavy daily traffic, supplied with direct current at 1500 volts, traverses the field, dividing it into approximately equal halves.

The first exploitation well at Lacq was completed in 1953 and since that time, no signs of casing failures have been noticed.

In view of the economic importance of Lacq Field, it was imperative that serious thought be given to early protection of the casings, so from the outset, a study was directed by A. M. L. Cauchois, technical adviser to general management of Societe Nationale des Petroles d'Aquitaine, assisted by J. R. Callais, chief design engineer to Societe Nationale des Gaz du Sud-Ouest. Recently the advice of Jack Barrett of Pan American Petroleum Corporation was solicited.

This study has been carried out according to standard techniques, numerous articles on which have been published in CORROSION. However, owing to the preponderance of currents varying with time, the methods have been adapted to meet requirements at the field.

Investigation of Currents on the Casing

The casing program at Lacq is: 13⅜ inches from 500 to 1200 meters, 9⅝ inches from 3000 to 3500 and 7 inches from 3500 to 4200 m. The 9⅝ inch casing, which has the greatest contact surface with the earth, is the chief concern of these investigations.

Means of Current Detection

The normal method of surveying the casing potential profile has been used. This method was first tried out in wells filled with mud but the unreliability of the measurements necessitated replacing the mud with crude oil, which is a better dielectric. The geology is such that the formation may be considered constant for distances of about 10 meters, consequently a spacing of 25 feet for the feelers of the survey instrument was adopted. A scraper was run in the casing before each log of the profile, which was made just after the casing was cemented.

Currents Affecting Casing

The electric field may be considered as the combination of currents from two sources:

1. A permanent current, constant in respect to time, or subject to little change. Moreover, this current is the combined result of ground cells and possibly of soil polarization due to the electrochemical changes caused by railway currents.

2. A temporary current directly proportional to the volume of railway current, which constantly fluctuates.

Because time appears as a new factor, the classic casing potential survey method no longer permits determining the electric state of the casing at a given moment.

Investigation of Permanent Currents

Permanent currents must be measured without traffic on the railway, or at least, during periods of low charge, so that the temporary fluctuations are negligible compared to permanent currents. Because the potential profiles generally were taken during periods of normal traffic, the logs are sometimes markedly irregular, conse-

* New international prefix "tera" = 1000 million.

★ Revision of a paper titled "Cathodic Protection of Gas Well Casing at Lacq Field," presented at a meeting of NACE Technical Unit Committee T-1 on Corrosion in Oil and Gas Well Equipment, Chicago, Ill., March 19, 1959.

quently the general form only of the profile should be considered in interpretations.

Figures 2 and 3 show the potential profile logs for two wells: Well A situated in the Eastern part of the field and Well B in the West (see Figure 1).

The profile of Well A indicates a zone of entry current down to the bottom of the 13⅜ inch casing at 800 m, a zone of low current exchange from 800 m to 2650 m and last, a strong current leaving from 2650 to 3000 m. Average density of the current leaving is 9.7 milliamperes per sq. m.

The profile on Well B is more progressive. The current entry zone appears down to a depth of approximately 900 meters. The current leaving then flows from the casing over a large surface extending from 900 m to the bottom of the 9⅝ inch casing. Maximum density of the current leaving is about 2.9 milliampers per sq. m.

Investigation of Temporary Currents

With the introduction of the time factor it is no longer possible to determine the potential profile at a given moment using classical method. It is therefore necessary to proceed in two steps:

1. The natural potential profile is logged when there is no railway traffic. Distribution of the permanent currents along the casing and the localization entry and departure zones is thus obtained.

2. The logging apparatus is left at a fixed point in the well and the potential variation is registered in terms of time during a period of variable traffic. This fixed point is generally at the top of the Albian formation where the current flow is at its maximum.

At any given moment the profile can be found approximately from the natural potential profile, without traffic, by a simple translation, the value of which is defined by the variation of ΔV at the fixed point.

Curve 1 of Figure 4 shows the current variation at a point in the casing of Well C when a train drawing 1000 amperes runs on the railway line. The vector translation at a given moment corresponds to the ordinate of this curve reduced by the original ordinate when no trains are running. Curve 2 expresses the correlative evolution of the rail ground tension at the place on the railway line nearest to the well and so proves the direct relation between the railway charge and the current flow in the casing.

Curve 1 of Figure 5 shows the natural potential profile of Well D without traffic. Profile 2 was logged when the railway charge was intentionally fixed (A train used 1000 amperes at a place nearest to the well). This second profile can be approximately deduced from the first by a translation, the vector of which, having a dimension of about 50 microvolts, is found from a curve similar to that in Figure 4.

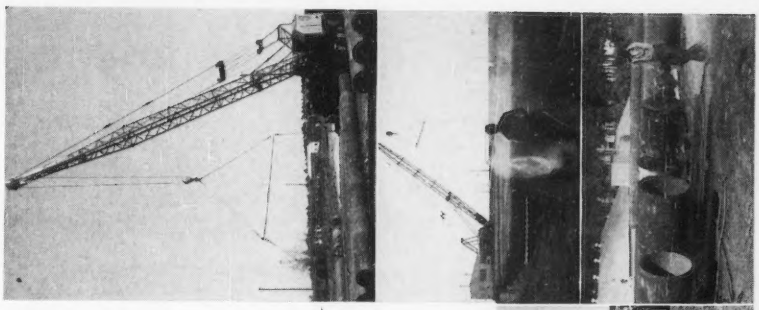
Cathodic Protection of the Casing

Because a means of protection must be able to eliminate both permanent and temporary currents the first task was to eliminate the permanent currents.

Criteria for Cathodic Protection Used

An attempt was made to define the optimum intensity of cathodic protection by the break point method. Owing to the depth of the wells, the presence of strong depolarizing environments in the Albian formation and the electrical instability of the ground, this method was at first a

(Continued on Page 16)



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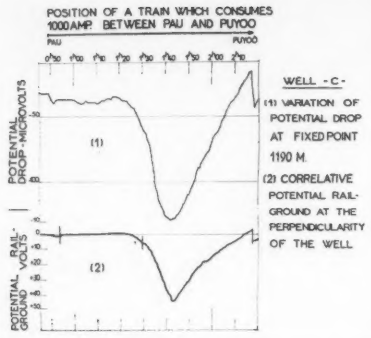
POSITION OF A TRAIN WHICH CONSUMES 1000 AMP. BETWEEN PAU AND PUYOO
PAU
PUYOO
POTENTIAL DROP-MICROVOLTS
WELL -C-
(1) VARIATION OF POTENTIAL DROP AT FIXED POINT 1190 M.
(2) CORRELATIVE POTENTIAL RAIL-GROUND AT THE PERPENDICULARITY OF THE WELL
POTENTIAL RAIL-GROUND VOLTS

Figure 4—Correlative potential of casing and railway on Well C.

Lacq Field—

(Continued From Page 15)

failure. These difficulties were overcome by acting on the polarization time, by making measurements systematically and by proceeding when the rail traffic was at a standstill. Figure 6 gives the polarization curve of Well B. The optimum intensity for cathodic protection is about 16 amperes. The potential profile of Figure 3 confirms the effectiveness of such a flow.

However, in view of the difficulties experienced in making and interpreting polarization curves, impressed current values generally were determined by analyzing potential profiles (see Figures 2 and 3).

It was also determined that intensity of the protection current by unit area of casing was between the generally accepted value of 5 to 20 milliamperes per sq. m.

Protection Against Permanent Currents

Separate values have been sought for each well.

The intensities applied vary from 15 to 50 amperes according to the zones of the field. These flows correspond to the average current densities of 5 to 17 milliamperes per sq. m.

The instantaneous range of the protection is, for a given intensity, variable according to the part of the field considered. For example, the potential profile of Well A (Figure 2) shows that an intensity of 50 amperes would be required, that of Well B confirms that 19 amperes are sufficient and that of Well C reveals that good protection can be obtained with 20 amperes.

Application of such large amounts of current was not without difficulty. It was difficult to establish ground beds having sufficiently low resistances because the top ground surface consists of pebbles and soft water. A horizontal ground bed formed of 50 meters of pipe lying in a

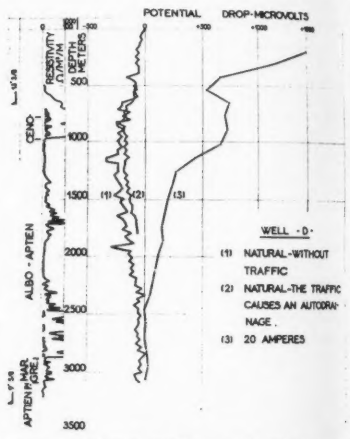

Figure 5—Potential profiles on 9⅝-inch casing on Well D.

conducting bed offers a resistance of 1 to 4 ohms. In some cases, it was necessary to drill 50-meter vertical holes to get below the surface bed of pebbles. In other cases, advantage was taken of the decrease of ground resistance created by a mud pit. These last methods allowed us to obtain ground beds with resistances of less than 1 ohm.

Generally, the amount of current was limited to avoid deterioration of the cement and possibly embrittlement of the casing.

The study of the current restrictions was followed by an investigation of the evolution of polarization in relation to time after application of cathodic protection.

Protection Against Temporary Currents

Lowering casing potentials by applying cathodic protection facilitates drainage through the ground of currents from the railway line. So, by diminishing the effects of permanent currents, temporary influences can be accentuated. Examination of that part of the danger attributed to each type of current, proves that cathodic protection results in considerable improvement.

In seeking for means of eliminating temporary currents it was supposed that they could be returned to sub-stations by

(Continued on Page 18)

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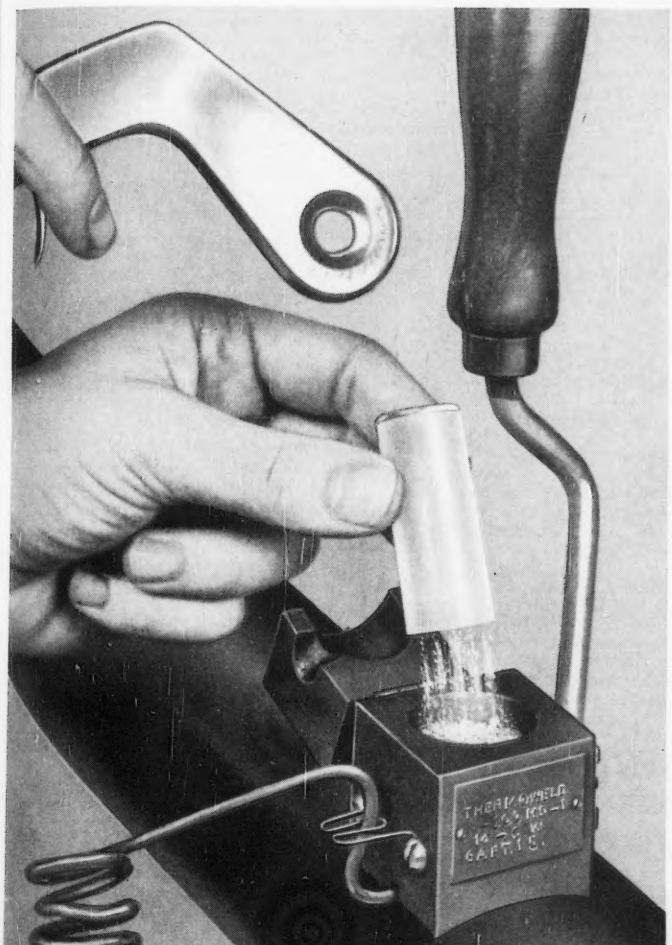

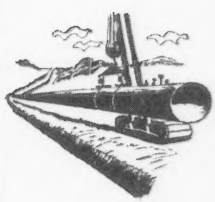

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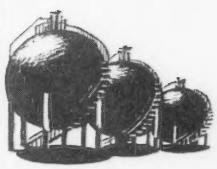

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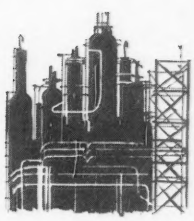

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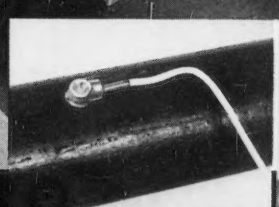

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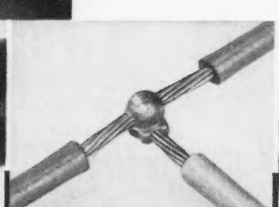

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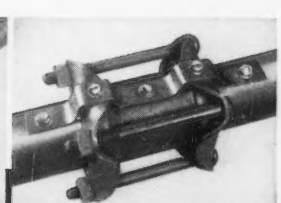

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Lacq Field–

(Continued From Page 16)

using low resistance path instead of the ground. It also was essential that the ground of the Lacq sub-station in the middle of the field be sufficiently negative.

The gathering system carrying the gas to the plant proved to be just the conductor required, but because the railway was hardly ever negative, even near the sub-station, drainage was impossible. Consequently, it will be necessary to adapt the sub-stations. A feasibility study has been undertaken with the cooperation of the S.N.C.F. (State National Railways). Two goals were considered: Lower the rail potential in the region of Lacq Field, and investigate ways of altering the electric installations to diminish return currents via the ground.

These aims led recently to a study of the possible effect of modifying a substation and even installing a new one on the field.

First results are encouraging and clearly indicate that modification of the means of supply is essential in reducing temporary currents. The remaining currents then can be eliminated by drainage. It was found

Figure 6—Current potential curve for Well B.

also, when the rail was negative to ground, that there is an auto-drainage effect which partially eliminates permanent currents and adds to cathodic protection (Profile 2 of Figure 6 was logged in the auto-drainage phase in the absence of cathodic protection).

Protection of All Underground Metal

All pipe lines in the gathering system are interconnected and electrically insulated at the well and plant ends. The pipe is coated with coal tar and glass wrap and protected by a combined system of drainage and cathodic protection using the rails as anode.

Special ring gaskets insulate the wells from all long distance surface pipes. In the near future they will be connected to the negative rail zone through the gathering system. The connection with this system will be polarized.

Estimate of Casing Coditions

Intensity of temporary currents seems to depend essentially on the distance from the well to the railway where the form of the permanent field varies directly with the geological zones penetrated.

Evaluation of permanent currents on the casing has been undertaken on only a few wells located mostly in the eastern and western sectors of the field. In each case, however, it was learned that while the current discharge is always in the Albian, its magnitude varies greatly from well to well.

These circumstances may be explained by relating the characteristics of the Albian formations to the electric log records.

The Albian formation varies considerably over the whole field. It exhibits very resistant reef facies in an elliptical zone, the center of which is Lacq. Within a radius of a few kilometers it develops into a shaly limestone then limy shale, to become shale only in the zone of Well B. On comparing the facies of the potential profiles, it is clearly observed that in the zones of shale facies the loss of current is continuous and progressive and that in the limestone zones, or those with intermediary facies, the losses are limited to the places of low resistivity.

The curves of Figures 2 and 3 illustrate this interpretation. In Well A which is drilled in the shale Albian, the loss is continuous. In Well B which penetrates the shaly limestone Albian, there is a low resistivity zone from 2700 to 3000 m, where the losses are concentrated. It is believed an investigation of the geographical site of the well and of the electric log will permit an approximate determination of the zones of probable corrosion and also a deduction of the intensity of the protection currents needed. The latter are proportionately larger as the facies contain more limestone, while the current drainage zone is to be found at a greater depth.

(Continued on Next Page)

Other Papers on Cathodic Protection

This issue, Page 97:
Performance of Zinc Anodes for Cathodic Protection in Sea Water, by L. J. Waldron and M. H. Peterson

This issue, Page 112:
Cathodic Protection for Metallic Water Tanks, by C. A. Bauer and L. C. Eddy

September issue, Technical Section:
Problems Encountered in Cathodically Protecting Pipelines Crossing Railroads, by F. L. Chatten

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Soil Microorganisms in Relation to Cathodic Protection, by J. O. Harris

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This general study has been conducted only for the 9⅝ inch casings. A potential profile log was run in the 7 inch to a depth of 4200 m. It showed that the volume of current exchange to ground appeared negligible.

Problems Yet to Be Solved

Potential profiles disclosed relatively strong current drainage zones and permitted calculation of their average densities. So far it is impossible either to calculate the expected life of the casing before it is perforated or even to affirm that there is a real danger.

What amount of current should be used for cathodic protection? Consideration of different classical methods led to an approximate value to be adopted.

Should this intensity be reduced after polarizing the casing over a certain period of time? The experiments carried out at Hassimessaoud in the Sahara, showed that with cathodic current of the same magnitude as that used at Lacq, the current flows at the bottom of the well might become very important. In fact, after a few days of polarization, this current flow may increase to several amperes, whereas it did not exist immediately after application of cathodic protection. Potential survey run after 8 days of polarization show that electrochemical changes provoked current departure zones (at least temporary) which are as great as those resulting from natural currents. A study of polarization phenomena in relation to the time factor is continuing.

How can the benefits of cathodic protection be increased? Investigation of the optimum distance between the anode and the casing showed that it would be suitable to adopt 100 meters.

During February 1959, an experimental coating of the 13⅜ inch casing was made with a coal tar epoxy resin and an isocyanate. If either of these prove helpful, they will be investigated further.

A means should also be devised for logging the potential profile at a selected moment so the time factor will no longer enter into calculations.

Conclusions

Adaptation of classical detection methods for determining the current in a casing has permitted recognizing at any given moment the probable distribution of currents therein and definition of the danger zones. Comparison of potential profiles and geological data led to attempts to extrapolate the results obtained on a few wells to cover the whole field and in this way to permit a general estimate of current conditions.

Cathodic protection permits combating the effects of permanent currents by employing intensities of 15 to 50 amperes. Temporary currents probably will be eliminated by modifying electrical supply installations and using as drainage conductor the gathering system.

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Suggested Methods and Practices for Cathodic Protection Coordination*

David T. Jones

Pacific Telephone and Telegraph Company
Los Angeles, California

Abstract

Discusses methods and practices used by a telephone company to coordinate its cathodic protection activities with other operators and owners of buried metallic substructures. This coordination is done to minimize effect of cathodic protection on metallic substructures. 5.2.1

Introduction

CORROSION ENGINEERS, until recently, were confronted with the problem of returning stray currents from direct current electric railways which flowed on metallic substructures back to the railway substation negative buss or through a suitable bond to the rail at a point removed from the substation.

To effectively drain these stray currents to their source required considerable cooperation and coordination of all owners of metallic substructures and the railway system operating people.[1] It further required that an operator of a metallic substructure not drain more current than necessary to satisfactorily restore the substructure potential to earth to a realistic value considering all other substructures which may be affected by the stray current situation. These requirements necessitated continual checking by all parties because of changes in the street car load or demands or because of additions, removals or rearrangements of the substructures in the area.

In most areas of the country and especially in Southern California, direct current railway systems are something of the past. However, a new problem necessitating coordination has been introduced by the use of cathodic protection by operators of metallic substructures. Due to industrial expansion and population growth in Southern California, there has been a constant increase in the number of gas, oil and water pipes and power and communication cables.

To provide an economic life to these mains and cables recently installed and to increase the life of some of the existing substructures, there has been an increased use of cathodic protection. There are about 700 cathodic protection installations in Southern California of which 110 are operated by the Pacific Telephone Company. This number of installations presents a great coordination problem.

This article discusses some of the procedures and methods used by the Pacific Telephone and Telegraph Company, Los Angeles Area, in coordinating the cathodic protection program with utilities, municipalities and other owners of metallic substructures for reducing or minimizing the effect of interference on foreign metallic substructures.

This article is not concerned with cathodic protection fundamentals or design. A pamphlet on cathodic protection is available from the National Association of Corrosion Engineers.[2]

★ Revision of a paper titled "Cathodic Protection Coordination Methods and Practices" presented at the Western Region Conference, National Association of Corrosions Engineers, Sep. 29-Oct. 1, 1959, Bakersfield, Cal.

THE PACIFIC TEL. & TEL. COMPANY — 1 OF 1

STATUS OF CATHODIC PROTECTION PROJECTS - REPORT NO. 12 — DATE 5-29-59

SINCE OUR LAST LETTER OF NOTIFICATION (REPORT NO. 11) DATED 1-22-59 THE FOLLOWING PROJECTS HAVE BEEN PROPOSED AS INDICATED IN THE "STATUS" COLUMN BELOW,

AREA LOCATION	RECTIFIER LOCATION	APPROX. ANODE LOCATION	NORMAL OUTPUT	DESCRIPTION OF PROTECTED STRUCTURE	STATUS	DATE
	1959 L.A. COUNTY THOMAS BROS. ATLAS GRIDS			BARE LEAD SHEATH CABLES		
VAN NUYS	A W/O SEPULVEDA & N/O VENTURA BLVD. (PAGE 22 C 3)	A W/O SEPULVEDA & N/O VENTURA BLVD.	32 V 8 A	ON VENTURA BLVD.	PROPOSED	7-15-59
GLENDALE	A W/O BRAND BLVD. & S/O DORAN ST. (PAGE 25 C 3)	A W/O BRAND BLVD & S/O DORAN ST.	48 V 4 A	ON BRAND BLVD. & ON DORAN ST.	INSTALLED	3-20-59
VAN NUYS	E/O DENSMORE ST. N/O VENTURA BLVD. (PAGE 22 B3)				TO BE REMOVED	7-15-59

D. T. JONES
SUPV CONSTR. FOREMAN

Figure 1—Typical form used for notification of intent to install, installation and/or removal of a cathodic protection system.

First Trial Tests

If test results of a preliminary corrosion survey indicate that remedial action in the form of placing cable or cables under cathodic protection is required, then a suitable ground bed or anode location is sought. Many factors are taken in consideration for the anode location such as (1) availability of 120 volt a-c power supply, (2) readily obtainable right of way, (3) lowest soil resistivity at location of highest cable-to-earth potential (lowest negative value to a copper-copper sulphate half cell reference electrode), (4) proximity to the cable run to be protected, usually not less than 150 feet away, if practicable, and (5) maximum practical separation from all foreign metallic substructures. In a congested area the desired separations cannot always be met.

When a cathodic protection system is being planned, pipe locators and substructure records are used to determine if there are foreign metallic substructures which could be affected by the proposed cathodic protection installation. If such structures are found, corrosion engineers representing owners of these substructures are notified of the proposed trial tests and are invited to participate in a joint test. In a congested or built-up area, owners of individual parcels of property are not notified; however, due consideration is given to any known metallic substructures in these properties which could be affected. Some gas and water utilities and municipalities place insulated joints in their gas or water pipes. As a result, such mains with their large area of coverage through their respective networks are the major problem requiring coordination.

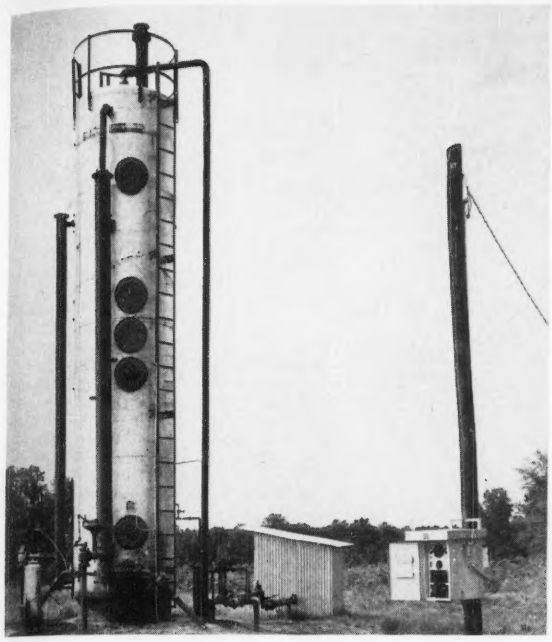
Trial tests are made with a portable d-c generator. Each test is made with various current outputs to determine the extent of the coverage on the telephone cable run. If representatives of the owners of the foreign substructures are present during the test, they also can determine the amount of interference, if any exists, from their test data. If they are not present, pipe to copper-copper sulphate half cell measurements are made for them with their permission.

From these test data, the size of the rectifier is determined which will give maximum protection to underground telephone cables and minimum interference to all foreign metallic substructures.

These procedures are used when conventional anode beds are to be installed, consisting of the common types of anodes buried at depths of not more than 20 feet.

In most cases where the telephone company is installing cathodic protection, corrosion engineers of the owners

(Continued on Page 22)



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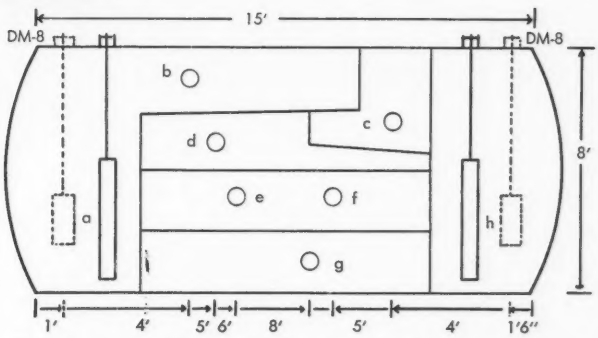

This drawing depicts anode placement for both magnesium and graphite.

ANODE	AREA	CURRENT
a	175 sq. ft.	1.75 Amps
b	100 sq. ft.	1.00 Amps
c	78 sq. ft.	.78 Amps
d	194 sq. ft.	1.94 Amps
e	97 sq. ft.	.97 Amps
f	97 sq. ft.	.97 Amps
g	154 sq. ft.	1.54 Amps
h	165 sq. ft.	1.65 Amps
	Total	**10.60 Amps**

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Suggested Methods—

(Continued From Page 20)

of the foreign substructures give permission to install the protective system with the provision that they make tests on their substructures at a later date to determine if interference occurs and to what extent. Usually these tests are made jointly.

Notification of Installation Plans

After these preliminary tests have been analyzed, the cathodic protection system is designed. Notifications of the proposed work then is sent to all interested parties. Figure 1 shows a typical notification giving all pertinent data concerning location of anode bed, rectifier location, structure to be protected, length, size and type of substructure (coated, wrapped or bare), proposed output of rectifier and tentative date of installation. By mutual agreement of the members of the Electrolysis Committee of Southern California, the page number and grid system shown on the Thomas Bros. Atlas for Los Angeles County is used to facilitate the approximate location of the installation. The page number and grid is shown under "Rectifier Location" on the notification. Other Atlases are used for other Counties. This notification is sent well in advance of the proposed installation date to enable owners of foreign substructures in the area of the proposed installation to analyze the condition of their plant and to suggest changes in our design, if necessary, to eliminate or minimize any adverse condition which could arise due to our installation of cathodic protection.

After the installation of the cathodic system, another notification is sent to companies and persons who received the notification of proposed installation; this one gives the installation date and rectifier output after the final cable to copper-copper sulphate half cell reference electrode measurements have been made on the entire section of cable plant being protected by the installation.

This same notification form is used also whenever rectifier output is increased by 2 amperes plus one-fifth of the preceding output due to installation of additional cables or changes for other reasons such as a change in location of rectifier or change in location or arrangement of the anode bed.

This notification procedure has been used satisfactorily for several years by members of the Electrolysis Committee of Southern California, the San Diego County Underground Corrosion Committee and other committees. This procedure has been recommended by the Correlating Committee on Cathodic Protection, a joint committee sponsored by American Gas Association, American Gas Association, American Petroleum Institute, American Public Works Association, American Water Works Association, Association of American Railroads, Bell System, Edison Electric Institute, International Municipal Signal Association, Inc., National Association of Corrosion Engineers, United States Independent Telephone Association and Western Union Telegraph Company.[3] The National Association of Corrosion Engineers has established Technical Committee T-7, "Corrosion Coordinating Committee" for the purpose of maintaining a centrally located source whereby information relative to various Electrolysis or Coordinating Committees throughout the country could be readily available to anyone desiring to know who should be contacted in connection with cathodic protection coordination. There are various sub-committees in the various regions in the country which maintain a closer relation within this smaller area. For the Pacific Coast, the sub-committee is T-7E. The location of the regional and subregional committees and their officers are given in CORROSION Magazine.

In practice, criteria to determine existence of interference normally is decided by the owner of the substructure which may be affected by interference.[4]

Allocation of Costs

Usually, each owner of metallic substructures assumes the cost of determining whether an interference problem exists on a substructure and assumes the cost of its own test work.[5] If the owner is a private party or a small organization which does not have a corrosion engineer or a corrosion engineer consultant, the owner company causing the interference generally covers the work by its own corrosion personnel to determine the extent of the interference, if any, and the necessary correction.

Joint ownership of a cathodic protection system has not been favored because the current amount required (on the average 5 to 10 amperes) to protect a run of telephone cable is comparatively small to the total required to protect the other metallic substructures in the area. Also, there would be complications in accounting procedures of capitalizing plant if the plant were sold to another party. Rectifiers are shared when other owners request current, if available, for protection of their substructures, or current is given in those cases where interference exists and drainage of current will eliminate or reduce interference.

The accepted practice by members of the Electrolysis Committee of Southern California and of the San Diego County Underground Corrosion Committee is that the interfering owner will provide sufficient current to the interfered owners at no cost to them for this current to restore the metallic substructure potential to a reference electrode to its original value. Any additional current required by the interfered parties to improve protection of their plant will be provided, if available, at a flat specific price per ampere year by the interfering owner. This flat price per ampere year has been derived from average costs, maintenance expense, tax and depreciation rates of cathodic systems for the past several years.

The time element between the installation notification given by an owner of a metallic substructure installing cathodic protection and the time when the interfering substructure owner initiates his tests for determination for interference is important. A reasonable period would be 18 months or less. The interfering party generally has spent considerable money and has attempted in good faith to locate the anode as far as possible from possible interfered substructures. In spite of precautions used, a mistake can be made. If the interfered party waits too long to make initial tests, factors such as a change in pipe coating or wrapping or even in the network arrangement of the substructure could affect the original situation. If after two years or more the interfered party makes a test and finds that interference exists, the Pacific Telephone Company even then will offer assistance and cooperation to control the interference.

Substructure Added After Cathodic Protection Installation

What should the rules be if an owner plans to place a metallic substructure in an area where there is already a substructure under cathodic protection?

(Continued on Next Page)

Novel Distributed Cathode Systems Are Used on Telephone Cables★

WHILE DISTRIBUTED cathode systems have not been commonly used for cathodic protection of cables in Southern Bell Telephone and Telegraph systems, some installations have been tried. One installation was made in Miami about 10 years ago in which the aluminum jacket of a polyethylene jacketed cable was used as an insulated conductor to distribute current over a section about a mile long. The ground bed was located in the Miami river.

Resistance bonds were located in alternate manholes and the current adjusted to give an even cable-to-earth potential over the whole distance. The installation performed well for a short time until the polyethylene sheath started cracking and the cable had to be replaced. The cathodic protection system did not cause the polyethylene to crack. The polyethylene sheath which cracked was an experimental material. Polyethylene used now has given satisfactory service as cable sheath.

Most of the replacement cable had plain lead sheath so 50 idle cable pairs in the sheath were used to distribute the current.

Because cable pairs and duct space are expensive, their use in installations of this type generally is not feasible. However, there are a few instances in Jacksonville, Florida where they have been used economically to distribute small amounts of current to protect toll cables.

★ Extracted from a paper "Cathodic Protection Installations for Lead Sheathed Cables," by Douglas T. Rosselle, Southern Bell Telephone and Telegraph Co., Atlanta, Ga., at a meeting of Southeast Region, National Association of Corrosion Engineers, Jacksonville, Florida, Oct. 1-2, 1959.

Pacific Telephone Company believes that, if the owner of the first structure installed elects to cathodically protect it from the time of installation and if proper notices of this intent and of the installation have been given, the second party should design for maximum separation or for coatings or wrappings on his structure.[6] In other words, the owner second in time, should provide his own protection or obtain current, if available, from the owner of the first structure on a pro rata cost of rectifier current output. These problems demonstrate the importance of giving notification of intention and installation.

Usually, the easiest and simplest means of correcting interference is to install a bond between the interfered metallic substructure and preferably the source of the direct current of the interfering metallic substructure. Amount of current drained will be that necessary to restore the potential of the interfered substructure to the potential which existed prior to installation of the cathodic protection system.[7] This current will be furnished without cost to the interfered party. In practice the cost of placing the bond from the interfered metallic substructure to a mutually suitably located connection box will be assumed by the interfered party. In practice the cost of placing the bond from the connection box to the interfering metallic substructure will be borne by the interfering party.[8]

Another method which has been used is to reduce the output of the rectifier until the owner of the interfered substructure agrees to a value which will provide to him an acceptable substructure to soil potential.[9]

In unusual cases in which the interference could not be corrected by bonds or by reduction in power output, the anode location was changed to one which did not create an excessive interference problem. Cost of this relocation was assumed by the telephone company.

These allocations of costs are not applicable to correcting interference brought about by the interfered party having placed his metallic substructure without protection after the interfering party's installation with cathodic protection.

It is the telephone company's practice in this area to place all cable entrances to subscribers' buildings served directly out of the underground cable run with insulated joints installed in them in order to isolate the telephone plant from water and gas mains.

In order to reduce the additional need for cathodic protection, the greater portion of all underground cables being placed today have non-metallic sheaths.

It has been mentioned previously that the "conventional" shallow anode produces a complex problem with many ramifications; however, it is now planned that all cathodic protection systems designed by the telephone company for future installation will incorporate the use of deep anodes at some 150-200 foot depths. From experiences of other owners throughout the country, the test data and experience gained so far indicate that the use of deep anode installations have greatly reduced and, in some cases, completely eliminated interference.

Conclusions

Even though cathodic protection does produce some complex problems, they can be resolved by using good engineering design and mutual cooperation, such as notifying all parties who could be involved of the intent to install cathodic protection and advise them of any changes or additions to the plant under protection.

To obtain the advantages of knowing of the physical changes in the various plants and to be aware of current changes in cathodic protection, all owners of metallic substructures operating networks should become members of a Corrosion Co-Ordinating Committee in the areas in which they operate.

Acknowledgments

The author wishes to express his appreciation to those corrosion engineers of other utilities, municipalities, irrigation districts and oil companies for their cooperation in resolving corrosion interference problems to mutually acceptable conclusions, thereby making it possible to present Pacific Telephone's experiences in this article, to L. C. Tupper, attorney, for his time and suggestions and to my wife, Kay, for her helpful suggestions and discussions.

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6. *Ibid.*
7. *Ibid.*, p. 23.
8. *Ibid.*, p. 27
9. *Ibid.*, p. 19.

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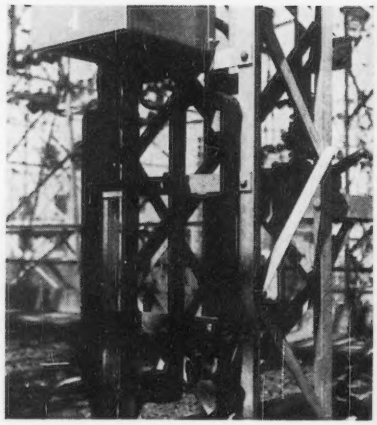

Figure 1—Low resistance, high current capacity resistor used in the shunt method of cathodic protection. The resistor is shunted between the cable conduit and the station ground network. (See schematic in Figure 2.)

Pipe-Type Cables Protected By Shunt Resistor System*

H. H. deLaneux and H. A. Medlock

Pacific Gas & Electric Company
Emeryville, California

Abstract

Discusses cathodic protection methods used by public utility company to control corrosion on pipe type underground cables and on lead sheathed cables in ducts. Describes shunt method of cathodic protection system used on pipe type cables. 5.2.3

★ Extracted from a paper titled "A Miscellany of Mitigated Corrosion Problems of a Public Utility" presented at the Western Region Conference, National Association of Corrosion Engineers, September 29-October 1, 1959, Bakersfield, Cal.

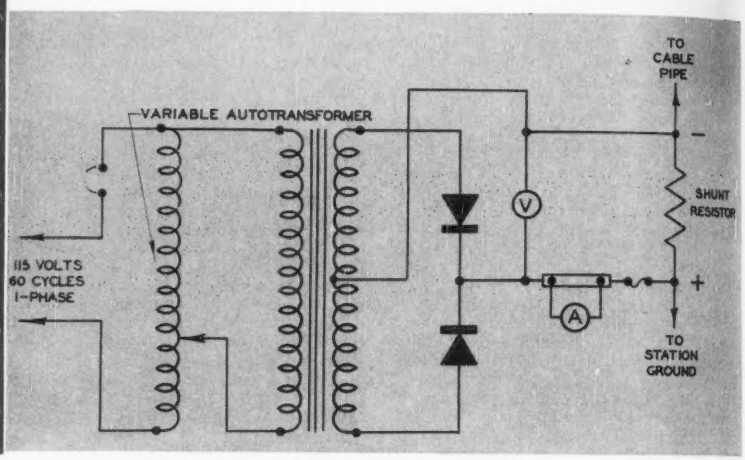

Figure 2—Schematic for shunt method.

UNDERGROUND CABLE corrosion is one of the more critical and more important problems which public utilities confront. This article describes the corrosion problem on pipe-type underground cables and on lead sheathed cables in ducts as experienced by the Pacific Gas and Electric Company.

Underground Pipe-Type Cables

A 110-KV pipe type underground cable system in San Francisco supplies the major power supply for the downtown area. Obviously, corrosion control on these important cables was carefully considered during installation and in later maintenance.

Because of their importance as San Francisco's major power supply and the difficulty of locating and repairing nitrogen leaks, a basic policy was adopted to the effect that corrosion leaks would not be tolerated.

The cables were the paper insulated, segmented type with a copper shield. Lines were pulled into a steel pipe (6⅝-inch OD diameter) coated with an asphalt mastic and filled with nitrogen maintained at 200 psi. The pipes were installed with care to avoid damage to the coating. Field joints also were asphalt coated. Before backfill was added, any holidays located by a high voltage spark gap test were corrected. After the backfill was added, conductivity measurements were taken in critical sections such as freeway crossings.

Use of an asphalt mastic coating was determined by awareness to environmental factors (tidal seepage, dissimilar metals in other underground structures and rubble deposited in fill operations) and case histories of nitrogen leaks on underground cables in the area. Since

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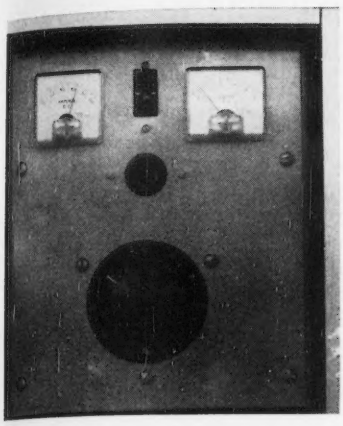

Figure 3—Variable auto-transformer used to control the primary voltage to achieve extremely fine control on the output current.

1949, annual tests have been conducted on these cables to measure and evaluate coating conductivity, to locate and repair any defects, to ascertain electrolysis conditions that might require cathodic protection changes and to eliminate any contacts with foreign structures.

These tests have indicated a progressive increase in coating conductance and a consequential increase in the corrosion hazard. This did not necessarily mean a reduction in the coating resistance but possibly additional mechanical damage to the coating. These tests also have disclosed galvanic currents established by interconnection of the steel conduits with the copper ground networks of the power plant and substations.

Cathodic protection of the pipe-type cables obviously was necessary where coating failures occurred. Selection of the method for applying cathodic protection to this cable system was a major problem because of the restriction that conduits had to be bonded solidly to copper ground networks at all cable terminals. The shunt method,[1] using an impressed current unit with an associated shunt resistor, was selected.

Shunt Method Used

The shunt method of cathodic protection provides an acceptable level of protection on the structure even though connected to the station ground network. Protective current is limited to a few milliamperes. Where low conductivity of the protected structure permits adequate protection with this small current flowing through the soil, there is little possibility of cathodic interference with other underground structures. This system, however, would not be used on structure having high conductivity coatings.

This shunt method derives its name from a low-resistance, high-current capacity resistor which is shunted between the cable conduit and the station ground network (see Figures 1 and 2). This resistor is designed to have a nominal resistance of 0.004 ohms and a current carrying capacity adequate to handle the maximum predicted fault current of the conduit. The resistor can be fabricated of two parallel stainless steel bars, each ¼ by 2 inches in cross section and 12 feet in length. It can be formed into a "W" to conserve space and to increase its structural rigidity (see Figure 2).

A rectifier with a secondary rating of 100 amperes d-c is connected across this shunt resistor with the positive terminal tied to the station ground side. Secondary voltage of the rectifier needs to be only a volt or two but should be capable of adjustment in small increments. This can be accomplished by a variable auto-transmorner (see Figures 1 and 3) used to control the primary voltage, thereby achieving extremely fine control from zero volts to the maximum rating of 3 volts on the output.

In operation, rectifier output current which flows through the shunt resistor is adjusted to produce a potential drop across the resistor of 0.3 to 0.4 volts, thus establishing a negative potential on the conduit with respect to the station ground or to the conduit's environment. The conduit can be given a pipe-to-soil potential of —850 millivolts as measured to a copper-copper sulfate reference electrode. At no point on the structure does the protective potential exceed this value.

Rectifier units were required at the end of each cable—a total of 10 units. After installation, each unit was tested for polarity, control of output and general operation. With all rectifiers in operation, their output currents were adjusted to provide a potential drop across the resistor of 340 millivolts. Pipe-to-soil potential measurements were made at numerous points along each conduit and at three terminals.

Potentials measured at cable terminals are difficult to evaluate—even to accept as authentic. Because many foreign structures are close to these terminals, potential measurements not

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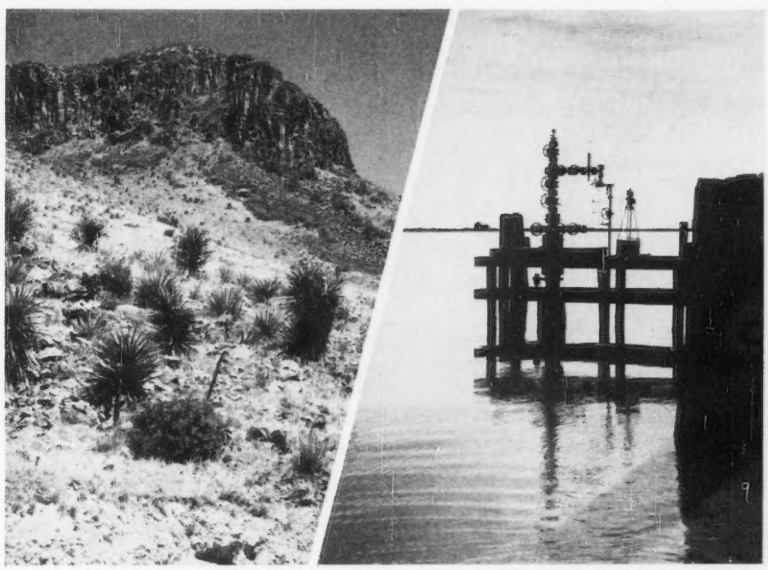

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Shunt Resistor—

(Continued From Page 25)

influenced by some potential field unrelated to the conduits are difficult to obtain. The transient or circulating currents are many times the magnitude of the current shown to be adequate by the isolated direct drainage test.

One method being considered to verify the adequacy of the protection level is placement of a reference electrode at a location remote to all conduits and referring all potentials to this remote placement.

Under some conditions such as an accidental contact with an infinite bare water system, the current discharged through the station ground network could increase to a value which could not be tolerated long. Because copper can go into solution at an annual rate of 46 pounds per ampere, protective current discharged through the ground network must be maintained at minimum values. If normal protection requirements are such that damage to ground network may result, a small cathodic protection station (or galvanic anodes) can be installed at some suitable location between the terminals. This unit's output can be adjusted to satisfy protection requirements of the entire conduit while the shunt installations maintain the proper potential of the conduit relative to the station ground. This auxiliary unit also would drain off transient currents, which is desirable, thereby eliminating one of the major causes of spurious data often obtained.

Future tests on the cables will include periodic checking of coating conductivity and cathodic protection requirements with the cables out of service and disconnected from station grounds.

Lead Sheathed Cables in Ducts

Corrosion failures on bare lead sheathed cables in 4-inch fiber duct lines necessitated replacement of several cable sections. These fiber duct lines were 4 or 12 KV three-phase feeder or tie lines, telephone and control cables. Bare copper neutrals were installed in the same duct as the cable.

To control the corrosion on these cables, cathodic protection was determined as the best method. Anode beds were not advisable because of the numerous underground metallic structures close to the ducts. These foreign structures would shield the ducts from pickup current unless it was introduced into the environments in some manner to avoid the shielding effect.

To solve the corrosion problem on one duct line which was permanently flooded by tidal waters, a string of high silicon cast iron anodes were installed in a spare duct. This spare duct was placed in a six duct run so that current distribution would be reasonably symmetrical. Graphite duct anodes were not used because of the possibility of sloughed off graphite being deposited in the duct. This graphite could seep through into other ducts, deposit on lead sheaths and create a galvanic cell.

Another approach was used to solve the corrosion problem on three duct runs. Some sections of the ducts were usually dry, other sections were almost always flooded and other sections varied between tidal inundation and rain water run-off. After these variable conditions were considered, it was decided that an anode bed would have to be installed at considerable depth to be independent of environmental variables and shielding effects of foreign structures. The anodes were installed at 40-foot depths. Because of installation difficulties at this depth, direct burial without backfill was specified. High silicon cast iron anodes were used instead of graphite because they are more rugged, have less likelihood of gas blockage and are believed to have a lower electrochemical equivalent when installed without backfill.

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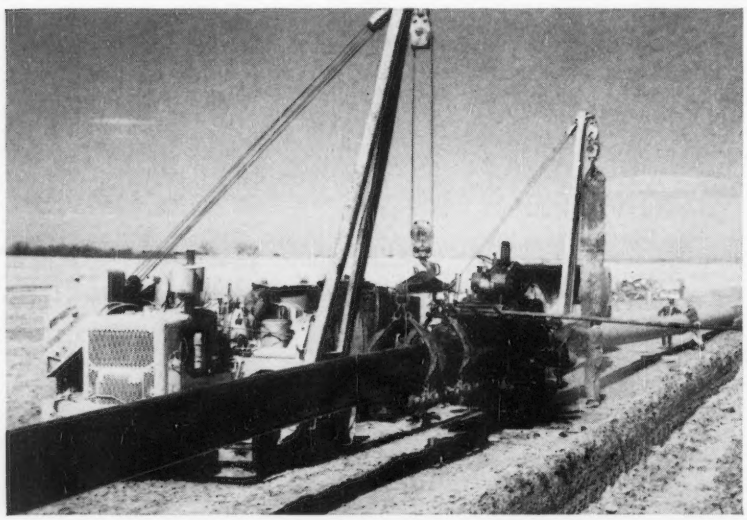

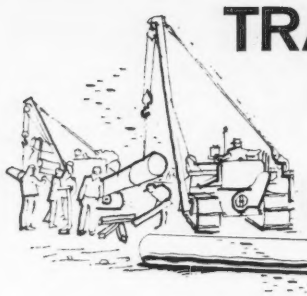

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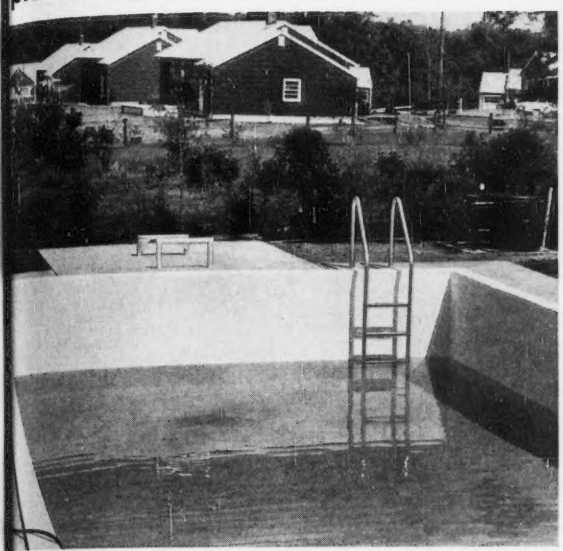

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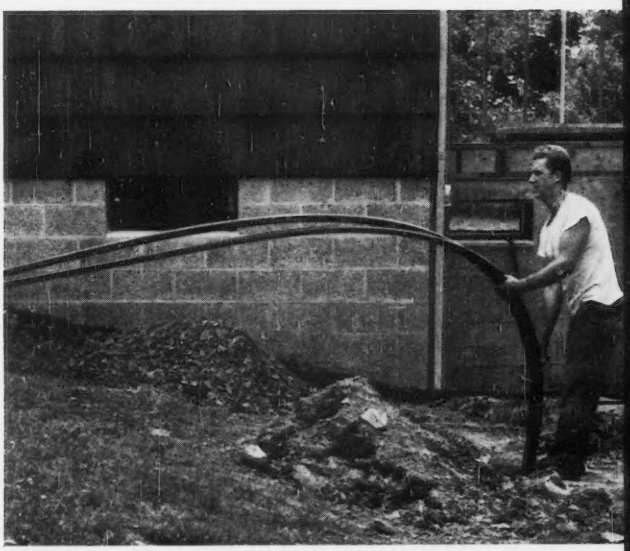

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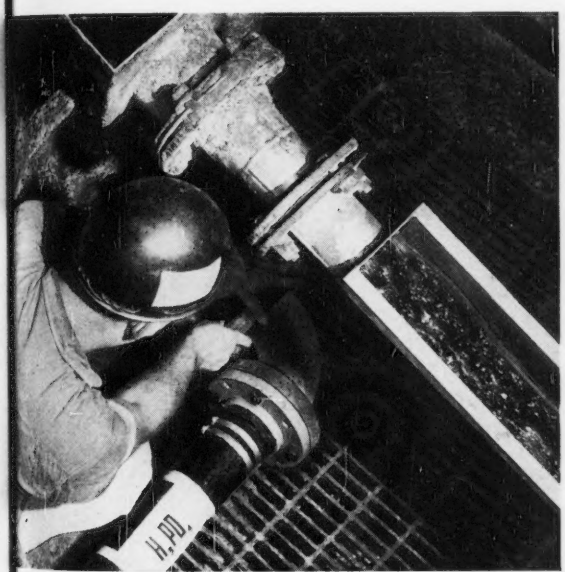

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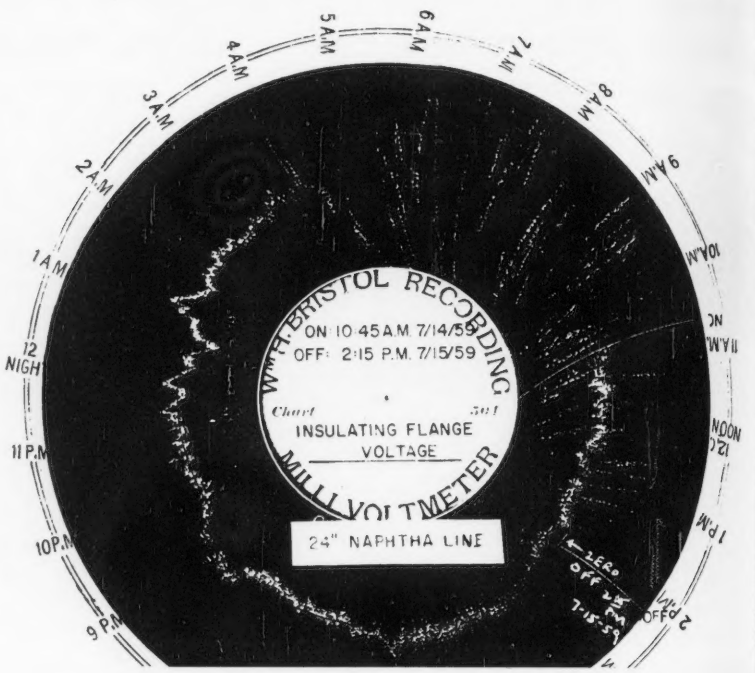
Sunspot Activity Correlates With Pipeline Voltages*

RANDOM OCCURRENCES of unexplained voltages and currents of large magnitude on underground pipelines have been the subject of investigation, discussion and speculation among corrosion engineers for many years. These effects have generally been referred to as "anomalous" currents or voltages.

About six years ago, recording voltmeters were run coincidentally recording voltages across insulating flanges in completely different pipeline systems over 250 miles apart. During the majority of the 24-hour period of the recordings there was no similarity between the pattern of the voltages traced by the two recorders. During a five-hour period on each recording, however, voltages increased radically on each recording and formed an identical pattern. This led to the belief that there was a relationship between the anomalous effects on pipelines and the variation of the earth's magnetic fields.

During the past summer, effects were noted that seem to put an end to speculation regarding the origin of the effects. On July 15, 1959, a "sunspot" activity seriously disrupted radio communication throughout the world. An AP news release in an eastern seaboard newspaper stated that the radio transmitting difficulty began at 4:00 a.m. Eastern Daylight Time.



The mysterious sunspots create magnetic storms in the inonosphere, which is that portion of the earth's upper atmosphere from which radio waves are bounced for long range transmission. These solar eruptions in July were considered by observatory personnel in Germany to be the largest in several decades. Officials at a Munich observatory estimated that one six-hour eruption had formed a crater the size of 50 earths.

The accompanying photograph shows a recording of the voltage across an insulating flange on a 24-inch naphtha pipeline. Note that from 2:00 a.m. July 14, 1959, to 4:00 a.m. July 15, 1959, the recorded voltages vary from about plus to minus 1.25 volts. At 4:00 a.m., a radical change occurs in the voltage and its pattern and values exceed both the positive and negative limits of the chart. Note that from 10:45 a.m. to 2:15 p.m., there is an overrun of the chart, with the voltages recorded on July 14 and July 15 being superimposed.

The "sunspot" activity continued for a period of days during which similar recordings were obtained. After "sunspot" activity diminished, voltages returned to their previous value. These phenomena were noted on other underground structures of unrelated systems.

The exact correlation of the disruption of radio communications and the change noted on the chart, seem to be ample proof of the origin of the "anomalous" voltages and currents.

★ Submitted by J. J. Meany, Jr., of A. V. Smith Engineering Co., Narberth, Pa.

See Page 18 for Other Papers on Cathodic Protection

PRIME and COMPETITIVE
Anti-Corrosion
PIPELINE MATERIALS

- Asbestos felt and Rockshield
- Glass Pipeline Wraps
  Mfd. by Owens-Corning Fiberglas Corporation
- Tapes
- Kraft Wraps
- Casing Seals
  Mfd. by Irish Pipe Line Supply Co.
- Alpon Internal Pipe Coating
  (Epoxy Coating)
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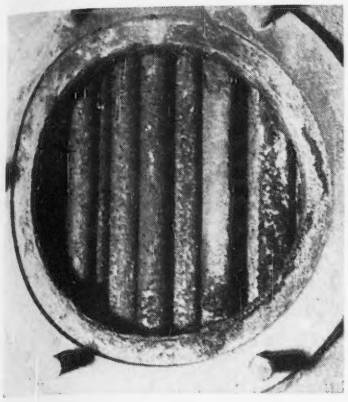
AINSLIE PERRAULT V. V. MALCOM

Soybean Processing Plant Stopped by Corrosion



PITTING AND STRESS corrosion from steam, hexane vapor and amino acids caused failure of these admiralty condenser tubes after only two years in a soybean processing plant.

Condenser Tubes Fail Twice From Combined Corrosive Attack

Steam, hexane vapors and amino acids were the combined corrosive that caused failure of condenser tubing in the solvent extraction system of a soybean processing plant.

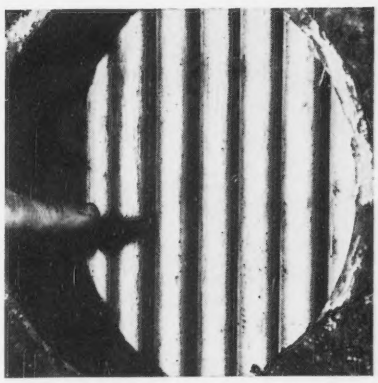
Built in 1951, the Funk Brothers Seed Company (Bloomington, Ill.) plant removes oil from soybeans by flaking and then soaking soybeans in hexane solvent to remove the oil rather than by the older expeller method which is primarily mechanical. The hexane process extracts 95 to 97 percent of the oil; the older method only 75 to 80 percent.

The plant processes over four million bushels of soybeans a day—about 400 tons. At this rate, any interruption to production can be costly.

First Corrosion Failure

First major interruption caused by corrosion occurred after two years' operation. Steam, hexane vapors and amino acids had attacked two upright condensers: a pre-evaporator unit containing 367 tubes ¾-inch OD by 10 feet long and a desolventizer-toaster condenser containing 880 tubes of the same size. The condensers recover hexane solvent after it has been evaporated from the extracted flakes and also from the oil-solvent mixture produced in the extractor.

Cooling water is circulated inside the tubing at 22 psi and at temperatures from 60 to 100 F. Externally, the tubing is subjected to hexane vapors, steam and amino acids circulating at temperatures of 200 F and above. Vapors enter the condensers from an 18-inch inlet about 3 feet up from the base of each vessel which stand 11 feet high. Tubing closest to the inlet had the severest corrosion attack, as seen in the photograph at the left.



STAINLESS STEEL Type 304 tubing shows no corrosive attack after more than 5½ years' service in a soybean processing plant condenser subjected to corrosion from hexane vapors, amino acids and steam.

In the original installations, condensers were equipped with phosphorized admiralty tubing. Baffles were installed to disperse vapors because initial inspections showed evidence of corrosion near the vapor inlets.

This admiralty tubing lasted only two years under the pitting and stress corrosion attack of the vapors. Vibration and thermal expansion and contraction contributed to the fatigue cracking of the tubes.

Corrosion Stops Production Again

After the first production break caused by corrosion, the failed tubes were replaced with annealed 85-15 red brass After two years' service, failure occurred again.

A careful analysis of the corrosion problem was considered necessary to prevent additional production interruptions. Sample sections of red brass, admiralty, 70-30 cupro nickel and stainless steel tubing were suspended inside an operating condenser for three months. Because of variations in stainless steel from one manufacturer to another, tubing made by several stainless producers were included in the test.

After the three month test, the samples were analyzed by a corrosion laboratory. Results showed that all tubing samples had been attacked by corrosion with the exception of Types 304 and 316 stainless steel. Type 304 was chosen for the condenser applications because it is cheaper.

Type 304 tubing has been exposed to the combined corrodents for 5½ years without corrosion failures, according to plant manager Delmar D. Walker. (See photograph above right.)

Two Steels Are Used To Solve Auto Oil Filter Corrosion

Use of two different steel materials were necessary to eliminate the problem of corrosion in automobile oil filters.

Replacement cartridges sold in service stations for automobile oil filters are subjected to corrosive conditions. The manufacturer experienced difficulties in trying to stamp necessary identifying data on the shell.

The answer seemed to be the use of steel black plate which could be enameled for corrosion resistance and easily painted with the data. However, the need for spot welding during assembly eliminated the possibility of an enameled metal because the enamel would act as an insulator. Also, the corrosion resistance factor would be affected by the welding heat.

The problem was solved by using two materials: one for the inner spool and another for the outer shell of the filter. Electrolytic tin plate was used for the inner spool, the component to be spot welded. Tin plate is not readily affected by corrosive substances. The coating is not so dense and cohesive but what it can be perforated, formed, rolled into tube shapes and welded.

The cartridge shell was made of steel black plate because it provides a suitable surface for enameling and imprinting.

Metallurgical engineering to solve the problem for Fram Corporation, Greenville, Ohio, was performed by Jones & Laughlin Steel Corp., 3 Gateway Center, Pittsburgh 30, Pa.

Technical Writing Course

An early fall course in technical report writing for engineers and scientists will be offered at the University of California at Los Angeles. Dates for the course are September 12-16.

Detailed information brochures can be obtained from Engineering Extension, University of California, Los Angeles 24, Cal.

Niobium Production to Be Achieved by New Process

A new process for continuous production of niobium (columbium) is being developed by Battelle Memorial Institute, 505 King Ave., Columbus 1, Ohio. Present chemical and electrolytic processes for niobium production is carried out on a batch basis.

Niobium may have possible use in future metallic nuclear reactor fuel elements.

The new process is a fluidized-bed vapor-deposition method. Hydrogen is used to reduce niobium pentachloride, which is fed into a reactor chamber containing a fluidized bed of seed niobium. Subsequent reduction by the hydrogen fluidizing gas at 1500 F produces metallic niobium which is deposited on the seed particles. Seed for the bed is replenished by grinding a portion of the end product and re-cycling it into the reactor.

RECORD and REPORT

BOOK NEWS

Transactions of the Vacuum Metallurgy Conference, 1959. Edited by Rointan F. Bunshah. 212 pages, 8½ x 11, paper. June, 1960, Associated College Press, 32 Washington Place, New York 3, N. Y. Per copy $7.50.

Twenty-two papers presented at the June 1-3, 1959 Conference on Vacuum Metallurgy sponsored by the Department of Metallurgical Engineering, New York University. Sections are: Vacuum arc melting and casting, vacuum investment casting, vacuum induction melting, vacuum degassing, electron-beam techniques in vacuum metallurgy and applications of vacuum metallurgy.

Workers concerned with exotic and high performance metals should find the data interesting. Considered, among others, are tungsten, titanium, zirconium alloys, beryllium, super-duty steels, refractory alloys, vacuum coating. A bibliography on application of electron-beam techniques in metallurgy is included. The book is liberally illustrated with line and halftone figures and is fully referenced.

Welding Handbook, Fourth Edition. Section Three Edited by Arthur L. Phillips. 512 pages, 6 x 9 inches, cloth. 1960. The American Welding Society, 33 West 39th St., New York 18, N. Y. Per copy, $9.00.

Special welding and metal joining and arc cutting processes are covered, including adhesive bonding and welding of plastics. Ultrasonic welding also is covered.

Chapters are included on forge, thermit, and induction welding; surfacing, metallizing, brazing, soldering, welding of plastics, adhesive bonding of metals, oxygen cutting, arc cutting, ultrasonic welding, welding by cold working and stud welding.

There are 335 illustrations and 78 tables. There is a 24-page alphabetical subject index and each section is thoroughly referenced.

Report on Marine Atmosphere Exposure of Galvanic Couples Involving Magnesium. by A. Gallaccio and I. Cornet. 26 pages, 6 x 9 inches. March, 1960. American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa. Per copy, $2.25.

Results of an atmospheric exposure study conducted for the Signal Corps Engineering Laboratories by the Ordnance Corps., Frankford Arsenal, Pitman-Dunn Laboratories. Objective was to acquire data on and evaluate performance of various protective systems applied to couples of magnesium with aluminum, cadmium, copper, steel, stainless steel and zinc. Protective systems include chromate conversion, anodic coatings, paint systems consisting of chromate primer and alkyd, phenolic and epoxy enamels. Pretreatments for steel were phosphate types and for copper and stainless steel, cleaning only. Exposure was near San Francisco Bay.

Tests showed alkyds offered less protection than phenolic and epoxy systems. Paint failures occurred more often on uninsulated than insulated couples. Anodic coatings reduced corrosion at dissimilar metal junctures.

1959 Supplement to AWS Bibliographies. Compiled by Edward A. Fenton. 32 pages, 8½ x 11, loose leaf, punched 3 holes, 4¼ inch centers. The American Welding Society, 33 West 39th St., New York 18, N. Y. Per copy, $1.50.

Articles classified under 28 topics that were published in the American Welding Journal, Jan.-Dec. 1959. Designed for integration with earlier bibliographies in the series covering articles published since 1937, this supplement brings the bibliography up to date.

X-Ray Absorption and Emission in Analytical Chemistry. By H. A. Liebhafsky, H. G. Pfieffer, E. H. Winslow and P. D. Zemany. 357 pages, 5¾ by 9 inches, cloth. John Wiley & Sons, Inc., 440 Fourth Ave., New York 16, N. Y. Per copy, $13.50.

Corrosion workers concerned with the important characteristics of film thicknesses, surface states, minor constituents and other information available through the use of x-ray techniques will be interested in this book. While it covers the subject exhaustively, it is designed also to be read in part by those who do not need to have comprehensive knowledge.

Among the chapter headings are: Origin and properties of x-rays, measurement of intensity, spectra and optics, determination of film thickness, x-ray emission spectography, reliability of x-ray emission spectrography.

Appendices include: Critical absorption edges and energies, approximate intensities of x-ray lines, wavelength characteristics x-ray lines, values of mass absorption coefficients of the elements in the region from 0.1 to 10 Angstroms, relation of wavelengths to diffraction angle for common monochromators, bibliography of element determinations and x-ray safety.

There is an author and subject index. Data are fully referenced.

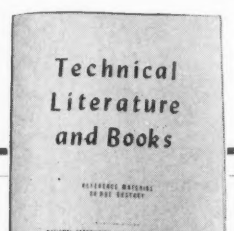
Symposium on Electrodeposition and Metal Finishing. Edited by T. L. Rama Char. 1960. India Section, The Electrochemical Society, Indian Institute of Science, Bangalore 12, India. Per copy, Rs 15.

Proceedings of the symposium held at Karaikudi, Dec. 27-28, 1957. Included are 36 papers covering many aspects of plating with chromium, cadmium, nickel, aluminum, brass, cobalt, tungsten, iron-zinc alloys, tin-copper, tin-zinc and uncommon metals; also data on electrolytic iron, electrodeposition of lead, various metallic powders, potential studies on zinc, anodic passivation of zinc, electropolishing of copper and stainless steel;

Hard anodizing of aluminum, corrosion and metal finishing, surface treatment of steel before painting, chromate treatments. In the section on corrosion and metal finishing the author estimates the cost of corrosion control in the United Kingdom and the United States.

New Polymers—New Problems. By H. F. Marks. 8 pages, 6 x 9 inches, paper. 1959. The 33rd Edgar Mruburg Lecture. American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa. Per copy, $1.

A consideration of some of the problems faced by the engineer seeking to use the new plastic materials, including mixtures of metals and plastics.



Over 1000 Articles In Corrosion 1945-60 Listed

Over 1000 articles published in CORROSION in the last 15 years are listed by subject matter. Each article includes the price for a copy, so you can select those in which you are interested and know exactly how much they will cost without further inquiry. Very valuable for research workers in the corrosion field. Complete through June, 1960.

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NACE Literature List Covering 1945-1960 Is Available Free

Ordering copies of technical articles printed in CORROSION will be facilitated through the use of the "Technical Literature and Books" list now available free from NACE, 1061 M & M Bldg., Houston 2, Texas. The document is a compilation of the titles and authors' names of more than 1100 articles published in CORROSION 1945-June, 1960 inclusive. The list includes an order number and a price for each article. Most articles are available as photocopies.

The articles are arranged according to subject matter under the following headings: Books, booklets, technical committee reports; general, testing, characteristic corrosion phenomena, corrosive environments, preventive measures, materials of construction, equipment and industries. The indexing follows the pattern of the NACE Abstract Filing Index. There are subdivisions under the headings: Preventive Measures and industries because of the large number of titles under these headings.

The book is paginated by sections so that additional titles can be added internally as they are published without disturbing the arrangement of the lists. Also, this permits distribution of parts of the whole for those whose interest is specific. For example, under Preventive Measures, the subdivision "Cathodic Protection" is designated 5.2 (same numbers as assigned in the NACE Abstract Filing Index). There are six pages under this subdivision, covering 163 titles. Coding is designed so that the year of publication is included in the number.

•

American consumers purchased five million television sets, half a million dish washers and 400 billion cigarettes last year.

At Westchester County's New Rochelle Sewage Plant:

Bitumastic coatings still sound after 5 years service on concrete tank walls

Opened in 1955, the New Rochelle Sanitary Sewer District plant is designed ultimately to serve a population estimated at 81,000 by 1975. In designing for the future, County engineers of the Department of Public Works recognized the problem of plant and equipment protection: that's why they specified that certain areas in the plant be protected with Bitumastic coatings.

On the settling tanks shown, two coats of Bitumastic® Super Service Black were applied to all concrete surfaces to prevent erosion at the water line. On trash racks and other intermittently submerged metal surfaces, the same coating system was applied. A recent inspection after five years of operation showed that these protected areas still require no recoating.

Bitumastic coatings were used on the inside of four concrete digesters, too. A base coat of thick, heavy-duty Bitumastic No. 50 covered with Bituplastic® No. 28, a coal tar emulsion-type coating, helped to seal the domes of the digesters against the corrosive effects of condensation.

Sanitary engineers in all states are familiar with the proved protective qualities of Bitumastic coatings. For a booklet describing their specific applications in this field, use the coupon. Koppers Company, Inc., Tar Products Division, Pittsburgh 19, Pa. District offices: Boston, Chicago, New York, Los Angeles, Pittsburgh and Woodward, Ala. In Canada: Koppers Products, Ltd., Toronto.

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New Device Inspects Equipment in Use By Ultrasonic Pulse

Internal corrosion of piping, tanks and other process equipment can be detected from the outside now while the equipment is still in service. Instruments and techniques developed by International Inspection, Inc., 1400 East Berry St., Fort Worth, Texas permit examination of erosion-corrosion of an elbow operating at 550 F with an accuracy of plus or minus 0.0025, for example. The new equipment is a hand-portable equivalent of the submerged supersonic testing systems now universally used in the aircraft industry.

Also, using the new equipment, it is economically practical to make 100 percent inspections of pipe and tubing, or other critical equipment to detect occlusions or flaws which cannot be detected readily by other non-destructive methods. Because the new device uses an ultrasonic pulse echo as its measuring tool, it has certain advantages over resonance-type instruments now extensively used for spot-testing. Inclusions and defects which measure as little as 4/64ths inch can be detected readily and with special care and equipment, defects as small as 1/64th inch can be seen. Defects are registered in an oscilloscope, or may be recorded on a chart for permanent reference.

Presently available transducers cover the full range of piping sizes from ¾-inch to flat plate. A special tool permits rapid 100 percent scanning of tank roofs or bottoms.

The equivalent of surmerged inspections is achieved by a hand-held transducer which permits introduction of a cushion of flowing water between the head and the material being inspected. In-service process piping up to 1000 F has been inspected, and there seems to be no practical temperature limit to the use of the equipment. There is also no limit to the thickness of metal that may be examined, nor to the kind, except that some cast iron cannot be reliably scanned. With a focused transducer pit depths can be measured. It is theoretically possible also to use the equipment for scanning non-metals, although the company has no experience in this application.

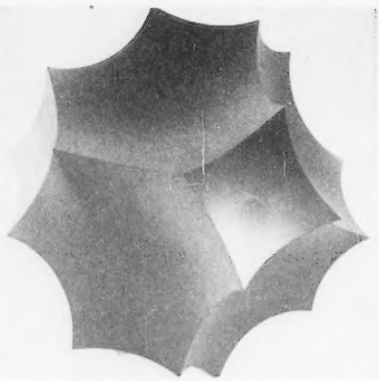
Reliable data are secured regardless of the material inside the vessel scanned. Smooth, adherent exterior coatings do not affect readings. Loose exterior scale or material must be removed from the surface.

International Inspection, Inc. is a subsidiary of Welex, Inc. The process is available on a service basis from any Welex or Halliburton office.

Plastics Conference Dates

"Plastics vs Corrosion" is the title given to a technical conference to be held October 5 at the Mark Hopkins Hotel, San Francisco, Cal. It will be sponsored by the Golden Gate Section of the Society of Plastics Engineers, Inc.

Additional information can be obtained by writing J. Wilfred Richardson, Rohm & Haas Co., 600 California St., San Francisco, Cal.



ALUMINUM MODEL, illustrating the momenta of conduction electrons, may give better understanding of the electronic properties of metals.

Unusual Model Helps Study of Electronic Properties of Metals

Better understanding of the electronic properties of metals may result from a model built at General Electric Research Laboratories, Schenectady, N. Y.

Shown in the accompanying photograph, the model illustrates the ways in which the momentum of a conduction electron varies as it travels through a metal.

The machined aluminum model has 14 concave faces. It serves as a physical representation of the hypothetical Fermi surface. As electrons move through the latticework of atoms, their momenta vary according to the direction they take. From one metal to another, the momenta vary depending on the number of atoms per unit volume and number of conduction electrons per atom.

In the model developed by physicist B. W. Roberts, these variations are represented by the varying distances from the center to the surface.

The model may give better understanding of such phenomena as the deHaas-van Alphen effect, cyclotron resonance, anomalous resistance and magneto-resistance properties.

Certain Coatings Increase Alloy Fatigue Strength

Coatings of certain polar organic compounds on specimens of steel, magnesium and copper-beryllium alloys markedly increase fatigue strength, according to recent National Bureau of Standards experiments conducted for the National Aeronautics and Space Administration.

Experiments showed that coatings of long-chain polar substances greatly increase fatigue strengths of the alloys named. On metal surfaces the polar compounds apparently form tightly packed monolayers that act as a barrier to water and oxygen molecules. These results are expected to have practical application in extending the useful life of certain metals and alloys.

Buoys Protected by Zinc-Dust Paints on Sandblasted Surfaces

Zinc-dust paints applied over a sandblasted surface proved best protection for buoys used as navigational aids in German coastal waters. Details of extensive tests involving zinc-dust paints from eight manufacturers, vinyl systems consisting of copolymerizations of polyvinylchloride and polvinylacetate; a combination of epichlorhydrin and bisphenol and a straight polyvinylchloride are given in a paper "Modern Methods of Buoy Preservation" by Karl Heinz Bohnsack, Waterways and Navigation Office, Tonning, Germany.

The paper is one of 150 to be presented during the Sixth International Technical Conference on Lighthouses and Other Aids to Navigation. The conference will be held in Washington during the week beginning September 26. A booklet containing abstracts of all papers may be obtained from Capt. J. A. Ciccolella, U. S. Coast Guard, Washington 25, D. C.

The paper on buoy preservation relates details of tests made since 1955, during which numerous schemes were tried in an effort to reduce the cost of maintaining the buoys. The North Sea area involved requires the use of 1100 buoys either in service or in stores. Adoption of a zinc-dust system including coatings in 200 micron (10^{-6}) thickness underwater; 250 near the top subjected to ice abrasion and 140 topside will produce an annual saving of about 25,000 DM per year.

Reinforced Plastic Buoys

Experience using polyester-glass laminates as spar buoys is related in a paper "Various Uses of Plastic Constructions," by Jorgen H. Meinich and T. Stranger-Johannessen, Royal Norweigan Navy. About 2000 buoys and spars are maintained in the sea by Norway. In addition there are approximately 4000 wooden spar buoys in reserve because when retrieved they require two years to dry in the depot. Iron spars are used to some extent also.

Beginning in 1956 experiments were started using plastic spars with such satisfactory results that about 100 polyester-glass buoys have been set out. Recently they have been filled with urethane foams to prevent sinking in case they are damaged in collision. Besides being virtually maintenance-free the spars do not weigh more than 40 kilograms, which makes them easy to handle.

Japanese Light Beacon Built

Installation of a steel-legged tripod lighthouse off the south coast of Honshu at Gozen Iza is described in another paper. "On the Construction of Gozen-Iwa Light Beacon," by Shinichiro Hirai, Maritime Safety Board, Japan, gives details of the $121,000 beacon, built in water 6 meters deep at low tide where maximum typhoon wave heights were calculated to be 7.8 meters high.

Cathodic protection was applied to the underwater portion of the structure and the legs above the water line insulated with concrete at a cost of $11,800.

Napko's Marcel L. Valois, Industrial Sales New Orleans, at actual job site.

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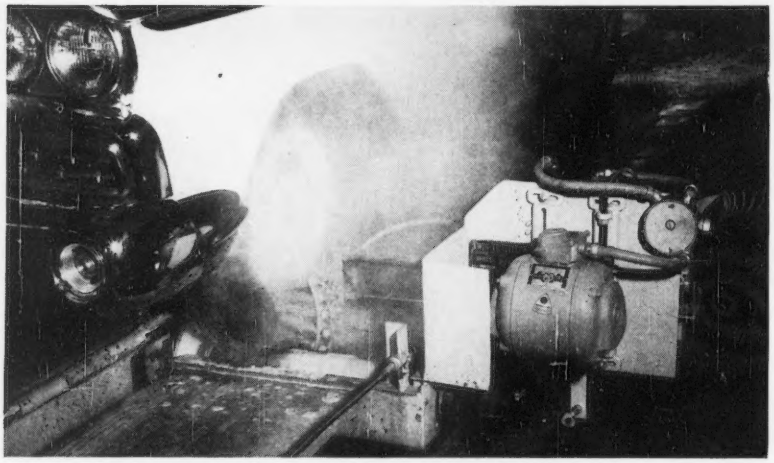


INTERNAL PROTECTION of components on a new electric motor design is achieved by coating components exposed to corrosive atmosphere. Expense of special protective enclosures is eliminated.

Electric Motor Parts Coated For Corrosive Exposure Service

Problems of corrosion control in electric motors and power drive assemblies to combat the effects of moisture, salt sprays, chemicals and other corrosive agents usually requires special protective enclosures, mounting and corrosion resistant paints and lacquers—protective measures that make the motors more expensive.

A new design in electric motors has been introduced to reduce this expense of special enclosures by internally protecting motor components. The new motor is designed to have full overload characteristics for many applications which previously required the use of expensive, totally enclosed motors. An application of the new motor is shown in the accompanying photography, made at an automatic car wash where the motor is exposed to water, vapors and residues washed from the automobile wheels.

The motor's stator coil encasement is given multiple applications of a special silicone sealing compound to achieve a resilient insulation with high dielectric strength. The coating is claimed to allow for normal pulsation of end coils under changing temperatures and loads to eliminate cracks and chips. Hot spots on the coil coating are avoided by maintaining a uniform thickness of material over the coil ends.

Heat dissipation is effected by through-ventilation design. Air can pass freely over, under and around the end coils.

Because the motor is not enclosed and vapors can pass through via the ventilation system, fan and rotor are coated with zinc chromate for protection. Motor leads are neoprene insulated and sealed by a weatherproof diaphragm. The terminal box also is sealed by neoprene gaskets and diaphragm.

Bearings are sealed for protection and longer service life. A cast iron bearing cap is used to protect the bearing from contaminants that could be blown through the motor from the fan end to the output end. Protection from external contaminants is provided by a grease reservoir and around the shaft by a labyrinth seal of neoprene to block passage of small foreign particles.

Called the Sterlicone Multi-Shielded motor and manufactured by Sterling Electric Motors, Inc., 5401 Telegraph Road, Los Angeles 22, Cal., the new motors are designed for applications involving excessive moisture, salt spray, oils, most chemicals and corrosive agents, dust, dirt and other contaminants.

Welding Society to Meet

The national fall meeting of the American Welding Society will be held September 26-29 at the Hotel Penn-Sheraton, Pittsburgh, Pa. Seventeen technical sessions are scheduled to cover the following topics: welded structures, resistance welding, brazing, electrodes and techniques, research and weldability, processes, non-destructive testing, and fabrication of weldments.

Water Pollution Conference

"Problems and Progress in Water Pollution Control" will be the theme of a national conference to be held December 12-14 at the Sheraton-Park Hotel in Washington, D. C.

The conference is being planned by Secretary of Health, Education and Welfare Arthur S. Flemming in response to President Eisenhower's recent directive.

ASEE Elects New Officers

Eric A. Walker, president of Pennsylvania State University, has been elected president of the American Society for Engineering Education. Vice presidents for the society are Melvin R. Lohmann of Oklahoma State University and Newman A. Hall of Yale University. Wendel W. Burton of Minnesota Mining and Manufacturing Company was re-named treasurer.

NBS Conducts Study To Eliminate Rapid Oxidation of Niobium

Niobium is potentially an excellent metal for structural applications in aircraft, nuclear reactors and other high temperature equipment because of its high melting point, strength even at high temperatures and ease of forming and machining. Unfortunately, however, this metal corrodes severely in air at temperatures as low as 400 C so that its usefulness in high temperature applications is limited at present.

To help solve this oxidation problem of niobium, the National Bureau of Standards is studying oxidation characteristics of the metal to determine the cause of rapid oxidation.

Previous investigations showed that two stages of oxidation occur: formation of an initial surface layer of niobium oxide at a rate which decreases with time and then a transition to rapid oxidation at a constant rate. The original oxide layer was thought to protect the underlying metal temporarily from further corrosion.

NBS tests have shown that the increased rate of oxidation is due to the continued nucleation and growth of porous niobium pentoxide, which causes a continuously refreshed surface of niobium metal to be exposed to oxygen.

Construction Begins on New Laboratory Site for NBS

Construction will begin soon on the first three building units of the proposed 20 buildings for new location of the National Bureau of Standards, U. S. Department of Commerce, Washington 25, D. C. The new laboratories will be located at Gaithersburgh, Maryland.

The first three buildings to be constructed will be an engineering mechanics laboratory, a radiation physics laboratory and power plant.

The new laboratories will provide a rural location where scientific programs can be undertaken without interferring in community life and without urban interference to NBS projects. The new location also will remove the NBS research work from the variety of mechanical, electrical and atmospheric disturbances present in a city and will reduce the effect of these forces on precise scientific measurements.

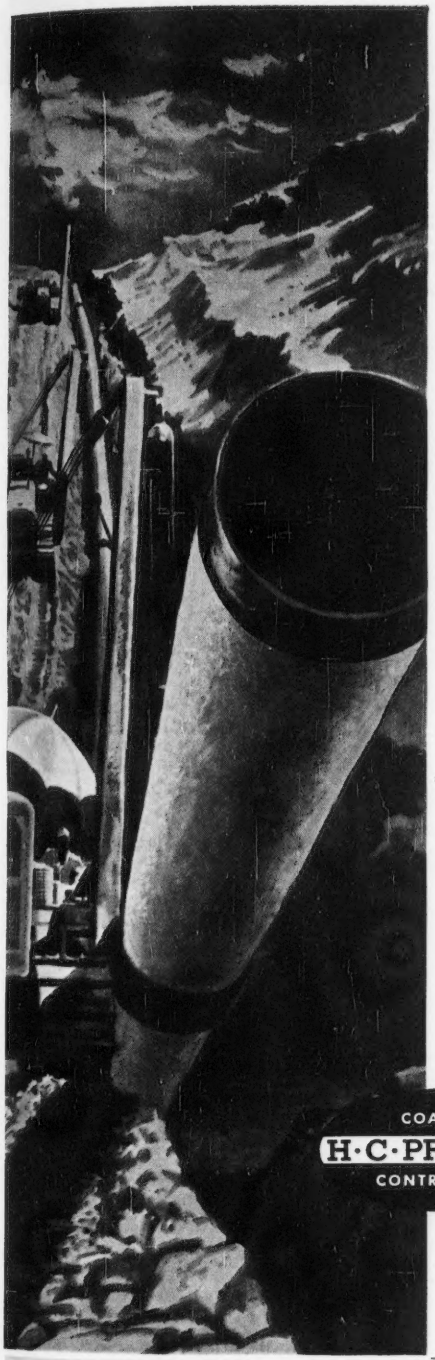
German Standards

A special department to prepare English translations of German industrial standards has been established by the German Standards Committee (Deutscher Normenausschuss). So far, 820 English translations have been made.

New lists of translations available can be obtained from the Beuth-Vertrieb GmbH, Berlin w 15, Uhlandstr. 175.

Environmental Terminology

A project on environmental terminology has been recommended to the American Standards Association by an ASA general conference held recently. People working in the rapidly growing field of environmental testing need a common language, according to the recommendation, to describe factors of environments.



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These tests prove conclusively that SOMASTIC® coating gives *maximum* protection against corrosion under the most adverse soil and moisture conditions. The result is longer line life, plus substantially lower maintenance and repair investments.

Initial cost? Modern manufacturing techniques of H. C. Price Co. permit application of superior SOMASTIC® pipe coating at *per-foot costs competitive with conventional pipeline coatings of all types.*

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AMCHEM Information Bulletin

Facts about pickling acid inhibitors

Nearly 40 years ago Amchem developed the first pickling acid inhibitor—RODINE—and with it revolutionized acid pickling. Only a few of the steel mills of the time believed that the addition of less than one percent RODINE to a sulfuric acid pickling bath could save substantial amounts of acid and metal, while increasing the life of the bath. Today there is no mill chemist who does not know about RODINE and its proven performance in eliminating waste of valuable metal and acid, and its ability to pay for itself through the savings thus gained.

In the past few years much attention has been focused on the use of wetting agents to accelerate pickling. But the fact remains that pickling cycles cannot be reduced by the use of wetting agents alone without severe overpickling problems. Wetting agents do help the acid run off surfaces removed from pickle baths, and, in so doing, reduce carry-over of acid into the rinse.

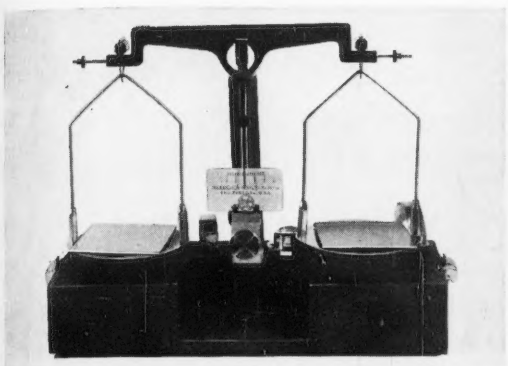
Wetting agents should not be confused with inhibitors to protect the metal and save acid wasted in pitting it. They should not be expected to miraculously speed the removal of scale, even at the expense of wasting the metal. However, the *proper* combination of *real inhibitor* plus correct wetting agent can have beneficial effects on pickling practices.

Only a true inhibitor such as RODINE can offer steel producers solid advantages in pickling. Baths can be operated longer and less acid is required to keep them at desired concentration. Costs of charging and cleaning the bath are reduced, less handling and storing of acid is required. A RODINE bath produces far fewer rejects—steel emerges with uniformly smooth, bright clean surfaces, free of pits, blisters and smut.

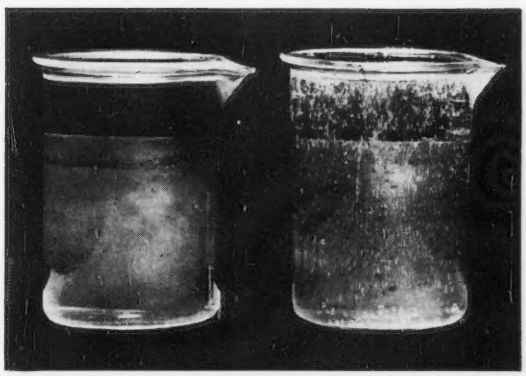
When an Amchem Technical Representative enters your pickle house, you can be assured of experienced assistance in improving your pickling. He will recommend the proper RODINE to 1) prevent needless consumption of acid after scale removal, 2) prevent smutting, pitting and overpickling, 3) produce a stable, free-rinsing foam blanket, 4) improve rinsability and reduce carry-over, and 5) produce a brighter, cleaner surface. Only the Amchem Representative can do this for you because he has at his command the most widely diversified and accepted line of real inhibitors and correct wetting agents available.

Since the early days of pickling acid inhibitor development, scientists in the Amchem Metal Protection Laboratories have constantly studied methods to make the RODINES stronger, more effective and more economical to use. The RODINES of today, as of 40 years ago, are in a class by themselves, have developed a reputation as *the international standard* in acid pickling inhibitors. The fruit of these years of laboratory research and experience in a highly specialized field, and the assistance of Amchem's trained field representatives—specialists in acid pickling techniques—are at your disposal for the asking.

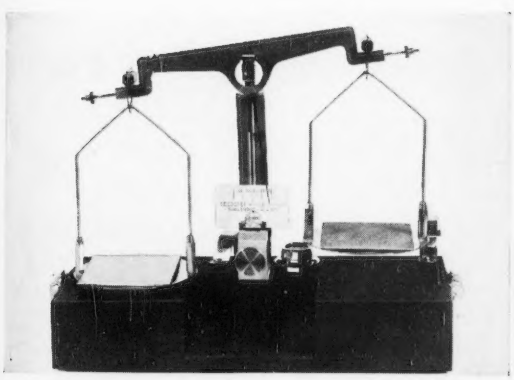
How Rodine Retards Acid's Attack on Metal



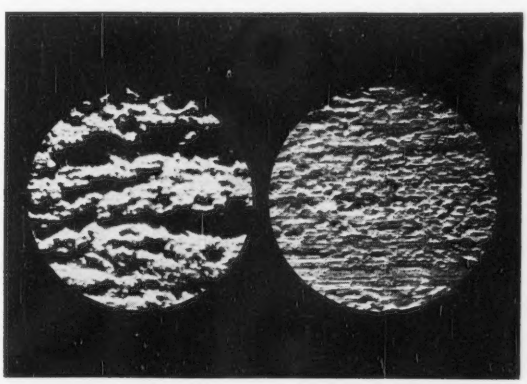
The photographs and microphotographs displayed on this page represent a graphic description of results demonstrating the effectiveness of RODINE in retarding the attack of acid on the metal. NO WEIGHT DIFFERENCE—two identical panels of low-carbon steel are shown being weighed here. They balanced the scale.



RODINE MINIMIZES ACID ATTACK—Each descaled panel was placed in a beaker containing 10% by volume of 66° Bé sulfuric acid of 160° F. The acid in the right-hand beaker was uninhibited. Acid in left-hand beaker was inhibited with RODINE 82, ¼% by volume of the concentrated acid. There was little or no hydrogen evolution in this beaker . . . RODINE retarded the attack of the acid on the base metal.



RODINE SAVES METAL—Both descaled panels were removed from the acid pickle solutions after 5 minutes. The one pickled in the uninhibited acid had lost 1.4% of its original weight. The one pickled in the RODINE-inhibited acid had lost only 0.001% of its original weight. This represents a saving of 26 lb. of steel for each ton pickled; and a simultaneous saving of nearly 50 lbs. of 66° Bé sulfuric acid when RODINE is used to inhibit the acid.



EFFECT OF UNINHIBITED ACID ON THE PANEL—This microphotograph of the low-carbon sheet steel pickled in the uninhibited acid shows deep pits and the crystalline character of the surface after 5 minutes in the solution.

EFFECT OF RODINE-INHIBITED ACID ON THE PANEL—This panel was pickled for 5 minutes in the RODINE-inhibited acid, then microphotographed. Only scale pockets and roll marks are visible; no pitting of the low-carbon sheet steel occurred.

AMCHEM RODINE

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AMBLER, PA. • Detroit, Mich. • St. Joseph, Mo. • Niles, Calif. • Windsor, Ont.

French Monorail Demonstration Indicates Advantages of System

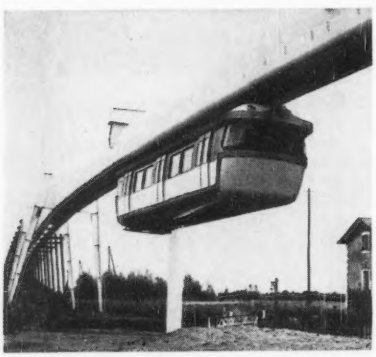
One possible answer to the ever-growing problem of city traffic may be the demonstration elevated monotrack built by French engineers at Chateauneuf-sur-Loire, near Orleans, France.

Designer and builder of the monorail system is Societe Anonyme Francaise d'Etudes de Gestion et d'Entreprises, a collaboration of 18 French enterprises including such companies as Renault, Alsthom and Michelin.

The society's first studies on the transportation system showed that underground space was scare and would be expensive construction. Surface areas were congested with streets, highways and other structures. Upward was the only alternative.

Steel Beam Used for Track

A hollow, electrically welded steel beam was used to obtain an economical track that was stable with high strength characteristics. It permits the thin concrete covered steel supports to be placed 97½ feet apart, with a ground encumbrance of less than three feet in diameter. Thus the supports could be placed in the middle strips between divided highways or next to sidewalks.



MONOTRACK DEMONSTRATION near Orleans, France, includes an aluminum car that handles 31 seated and 91 standing passengers. It travels at high speeds on rubber tired carriages housed in a hollow steel beam. Supports are required at 97½-foot intervals. (Photograph courtesy of France Actuelle Magazine).

The track has the advantages of hiding the bogies (wheels, supports and electric motors for power), protecting the rolling mechanisms from weather exposure, providing a muffler for the low noise caused by the rolling bogies and also providing a place for necessary power supply and signal lines.

Rolling Gear Used

The monorail car hangs from two bogies, each with four auto-size vertical wheels and four small horizontal guiding wheels which run against the inside walls of the track. All wheels are rubber tired as are some trains on the main subway lines in Paris.

Each bogie has two traction motors which develop 115 horsepower each. A steel rim prevents serious damage in case of a tire blow-out. A double brake system is used—pneumatic and rheostatic (action on the motors).

Aluminum Car Designed

The aluminum car being used on the French demonstration monorail is 57 feet long, 10 feet wide and 10 feet high. It has room for 32 seated passengers and 91 standing.

Suspension system joining the car and its bogies is a compressed air cushion.

Lightness of the car, traction from rubber tires and power from electric motors permit rapid acceleration, high cruising speeds and quick decelerations when necessary.

Safety measures for control of the system include a reproduction of line signals in the engineer's cabin. Safety would be important: cars would be passing a given point every 90 seconds in a city. The emergency brake goes on automatically if a signal is passed. The engineer also has constant telephone communication with the traffic dispatcher.

Estimated Cost of System

All-inclusive cost of this new monorail system is estimated between 2.5 and 3 million dollars per kilometer (about five-eighths of a mile). This compares with a cost between 14 and 20 million dollars per kilometer for conventional subway lines.

High Strength Alloy Obtained by Adding Fibers to Metals

A unique metallurgical process which combines ceramic fibers with metal to attain extremely high strengths at high temperatures is being studied for applications on space vehicles and aircraft.

Studies on the fiber-metal project began with the increasing demand for craft. Landing gears, fittings and pro- high strength alloys suitable for use in pulsion assemblies on these vehicles hypersonic airplanes, missiles and space- must resist intense heat, erosion and radiation.

Basic concept of the alloying method is similar to that in which a combination of glass fiber and plastic has been used for products ranging from glass fishing rods to rocket nozzles and cones operating at extremely high temperatures.

In these fiber reinforced plastics, strengths to 300,000 psi are possible—much beyond that possible with plastic alone.

A similar increase in structural strength would result in combining ceramics with a metal matrix, according to engineers at Horizons Incorporated, 2905 East 79th St., Cleveland 4, Ohio, the firm conducting research on this project. Some high temperature fibers now available for alloying with metals have strengths as high as three million pounds per square inch although metal matrix strength alone would not exceed one-half million pounds per square inch. Such substantial reinforcement of a metal structure is possible even though the structure is used at temperatures close to the metal's melting point.

A reduction in weight also would result from the fiber-metal combination.

Selection of Materials Studied

Search for an ideal combination of metal and fibers is requiring the examination of many alloys and ceramic materials. Research indicated the metal to be used should exhibit no precipitation phases resulting from alloy combination or heat treating and had to maintain oxidation resistance substantially to the melting point.

Selection of a fibrous material imposed similar strict requirements. The inorganic fiber must have a density less than that of the metal to be used, a melting point much higher than the metal matrix, a coefficient of expansion equal to or less than the matrix, an elastic tension modulus suitably greater than the metal and strength much greater at high temperatures than the metal under consideration.

An alloy which has come close to meeting requirements is an 80 percent nickel and 20 percent chromium composition. Inorganic fiber best suited for the combination is an aluminum oxide of sapphire or corundum single-crystal form. These tough, flexible fibers can be wrapped around a radius of a fraction of an inch without fracturing. About half the fibers' high tensile strength is retained at the melting point of the nickel-chrome alloy.

Several problems must be solved before a satisfactory structure can be produced for commercial or military use. Primary difficulties involve determination of fiber-to-metal ratios, fiber dimensions for optimum strength, techniques for wetting and bonding, and fabrication methods to maintain uniform dispersion of fibers in the matrix.

Electrochemical Meeting to Include Corrosion Sessions

Sessions on corrosion will be included in the October 9-13 meeting of the Electrochemical Society, Inc., to be held at the Shamrock Hotel, Houston, Texas. Other sessions will be on batteries, electrodeposition, vapor deposited coatings, semiconductors and electrothermics and metallurgy.

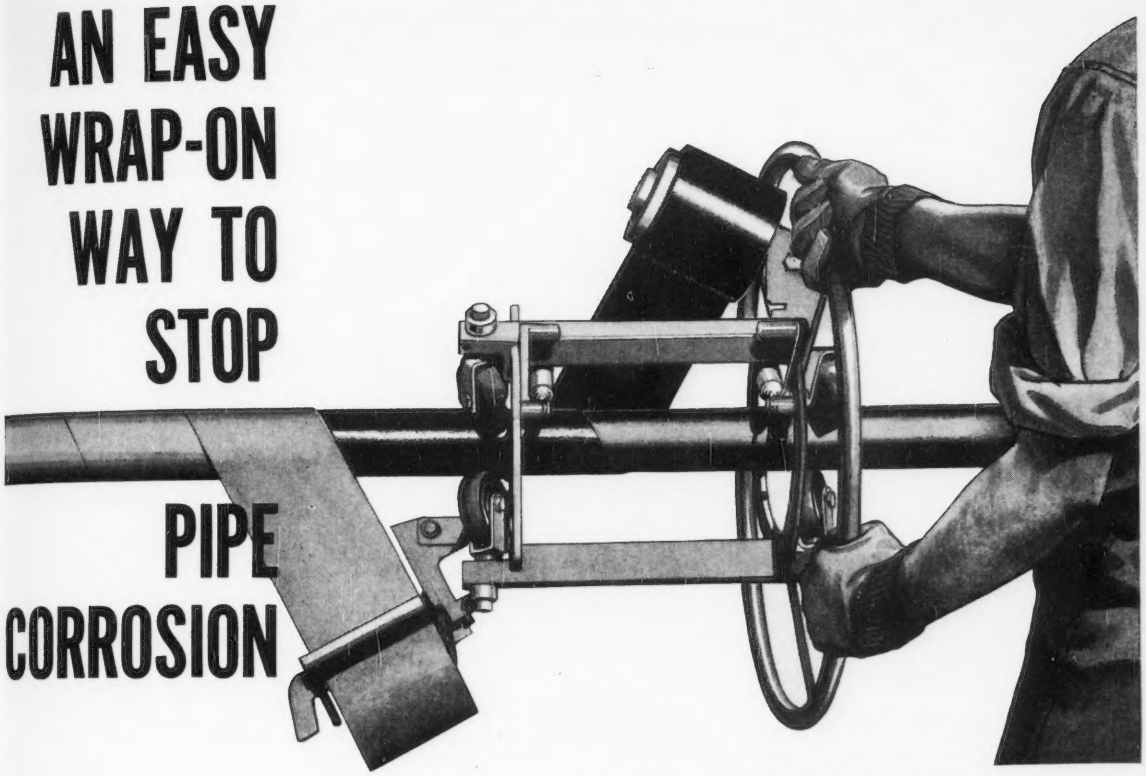
The corrosion sessions will be on can corrosion, oxidation, unusual valence states and the difference effect, certain properties of the interface related to corrosion, passivity, dissolution of metals and semiconductors and a general session on corrosion.

Navy Builds Laboratory for Measuring Magnetic Fields

A new facility for measuring low level direct current magnetic fields in ship components has been built at the United States Naval Engineering Experiment Station at Annapolis, Md. Primary purpose of the testing facility is for research and development of magnetic countermeasure devices and low magnetic field shipboard equipment to provide a means of rapid testing and analyzing of prototype equipment under running conditions before permanent installation in naval vessels.

The necessity of such magnetic countermeasures has been caused by recent advances in naval underseas warfare.

AN EASY WRAP-ON WAY TO STOP PIPE CORROSION



The new CORR-PREV Pipe Coating Team comes in easy to handle tape form. It goes on pipe faster and lasts longer than corrosion-proof paints. It's cleaner and simpler to apply, too, than hot tar coats. And most important, this new CORR-PREV Team offers all the cost-cutting, pipe-saving advantages of *polyethylene* tape.

With CHASEWRAP Abrasion-Resistant Overwrap and Chasekote polyethylene tape, you get the easiest and best pipe protection at *lowest applied cost!* There's no heating, drying, clean-up or shut-down time. Each roll is factory-uniform in thickness and quality. And all coating materials — including rental of high-speed applicators — are available from one reliable source. Want samples? Specs? Prices? Write **CHASE & SONS, INC., 26 Spruce St., North Quincy, Mass.**

Chasekote*

Pressure-Sensitive Polyethylene Tape has an extra-heavy adhesive mass that clings to clean pipe on contact . . . makes a perfect, permanent bond that rustproofs, waterproofs and insulates pipe against all types of corrosion.

Chasewrap*

Protective Overwrap is a tough, wrinkle-free outerwrap that shields against abrasion, wear, and physical damage to the tape. Wraps simultaneously with the tape. Superior to rag and felt wraps, yet far lower in cost.

**Trade name of Chase & Sons, Inc., long-famous for protective and insulating tapes for electrical wire and cable*

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FOR YOUR ELECTRICAL INSULATION PROBLEMS SPECIFY CHASE FRICTION, PLASTIC, RUBBER, NEOPRENE AND BUTYL TAPES

Cable Withstands 20-Year River Exposure

Tests Conducted to Check Performance Of Type 304 Stainless

Tests conducted on Type 304 stainless steel wire cable after a 20-year exposure from moderate to severe service conditions have shown no loss of strength, no corrosion and little abrasion.

The wire cable was used for daily raising and lowering of a 12-ton taintor valve mechanism at a lock and dam on the Mississippi River near Guttenberg, Iowa, about 225 miles south of Minneapolis, Minn.

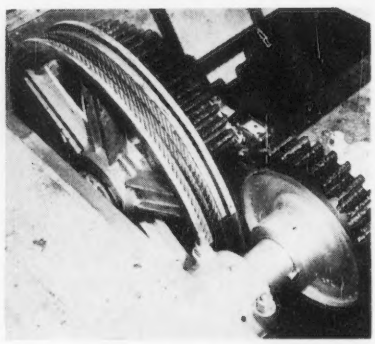
Twin cables were wound over two 54-inch diameter drums (see accompanying photograph) to actuate the valve. Cables were attached to the lower front corners of the valve. Pivot upon which the valve rotates was designed to carry part of the 12-ton load with each 1-inch diameter cable supporting an estimated 8500 pounds.

River water to which the cable was exposed contained moderate to heavy silt in suspension depending on the season of the year. Dissolved minerals and gases also were present to add to the corrosive conditions.

Reasons for Selecting Stainless

Three reasons were given by the U. S. Army Corps of Engineers for the selection of Type 304 stainless to be used for the wire cable. The Corps of Engineers is responsible for controlling water flow on the upper Missisippi River for flood control purposes.

First, stainless was chosen for its resistance to environmental damage. Four conditions were considered: (1) silted water and its contents, (2) climate variation with temperature extremes from —30 to 100 F, (3) long term exposure to drum greases and lubricating fluids needed for efficient hoist operation and (4) abrasion of outer wire layers during winding and re-winding of the cable on the drum.



STAINLESS STEEL WIRE cable exposed 20 years to Mississippi River water showed no corrosive attack. Extensive tests on sample sections also showed no loss of strength.

Second, stainless wire was selected for its strength. A load of 8500 pounds was considered minimum. Starting and stopping stresses created momentary loads much beyond the 8500-pound static pull. Acceptance testing under the original specification called for a breaking strength above 85,400 pounds, far in excess of that anticipated in service. Other materials could withstand this stress, but as corrosion proceeded a loss of strength would be a natural consequence.

Third reason for selecting stainless steel cable was the favorable strength-weight ratio of stainless alloys. Other materials capable of supporting the 12-ton valve for years would have required a more costly, heavy duty hoist mechanism because of cable weight and bulk.

Service Conditions Encountered

Service conditions that prevailed were as follows: (1) continuous submersion in river water with a straight pull of about 8500 pounds, (2) exposure to normally varying climatic conditions and slight flexing under the 8500-pound static load, (3) exposure to lubricants and greases needed for hoist operation and (4) repeated flexion under an 8500-pound load while stretched over a drum diameter of 54 inches.

Tests Used to Determine Performance

After the cable had been in service for 20 years, the Corps of Engineers removed three 15-foot lengths, representing each type of service condition, for extensive mechanical and chemical testing. Each rope sample was tested to determine its breaking strength, then unreaved and individual wires tested for appropriate mechanical properties. A section of each rope sample also received chemical analysis.

Breaking strength of the sample removed from underwater service was 91,050 pounds; that of the cable exposed to the atmosphere was 91,350 pounds. The section wound around the drum hoist developed a breaking load of 90,200 pounds. All these strengths exceeded the original acceptance specification of 85,400 pounds.

Gross appearance of each sample was normal. Some interstitial openings were noted in the sample removed from the water.

No internal wire failures were found in any of the samples. External wire failures also were absent except in the sample exposed to water. A crown fatigue type fracture was seen in one wire of one strand.

Abrasion was noted only in the sample flexed over the drum hoist. Crown wear in this sample was light, amounting to a 4 percent reduction in the original wire diameter.

On the submerged sample a white coating was found. Chemical analysis proved this coating to be a lime deposit. No evidence of corrosive attack was found on any sample.

First of Spectral Data Sheet Series Published

First in a proposed series of spectral data sheets on organic compounds has been published by the Manufacturing Chemists' Association, 1825 Connecticut Ave., N.W., Washington 9, D. C.

The first issue contains ultraviolet, infrared and mass spectral data to help industrial laboratories identify processing fractions and pure compounds. Later issues to be available at six-month intervals will contain Raman and nuclear magnetic resonance spectral data.

Data on spectral properties help in making rapid spectrographic analyses of plant streams and product materials in manufacturing processes and for identifying individual compounds in analytical and research work.

Information for the data sheets was compiled by the Carnegie Institute of Technology through a project sponsored by the MCA.

NEW Casing Seal!
WmSON "U" SEAL

MAINTAINS TIGHT SEAL EVEN WHEN DRAG SECTION CHANGES POSITION IN CASING

This versatile and flexible "U"-Seal offers NEW ECONOMY in casing seals.

The special demonstrator pictured shows the flexibility of the "U"-Seal. The pipe position changes, but the seal remains tight. A special fungus and bacteria retardent is added to the compound for extra protection. Seals available in sizes 2" and larger.

VERSATILITY OF INSTALLATION

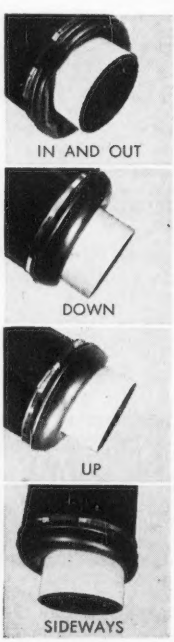
The fully reversible "U"-Seal is easy to install in the new "U" shape or in WmSon's familiar "Z" shape. A screwdriver is the only tool needed to tighten the two stainless steel bands.

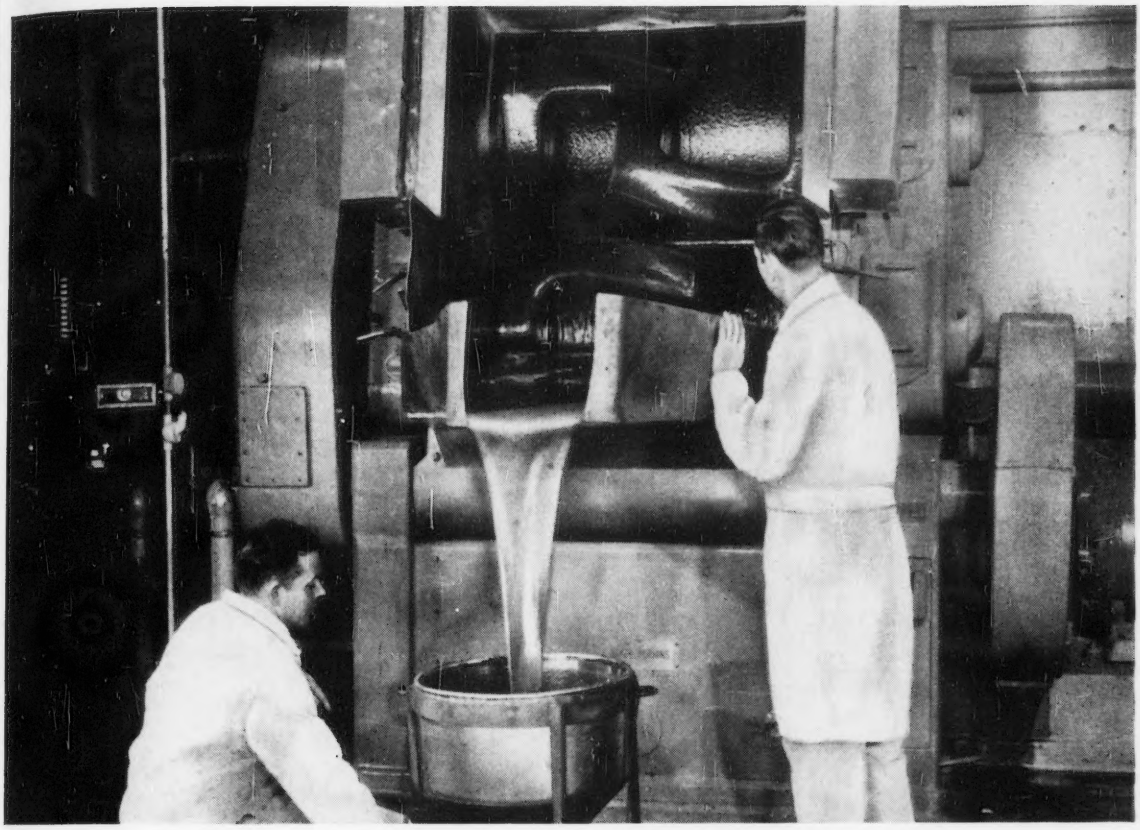
Write Dept. D for bulletin A-281

YOU CAN SEAL WITH A "U"-SEAL

T.D. Williamson, Inc.
P. O. BOX 40 TULSA 2, OKLAHOMA

REPRESENTATIVES AROUND THE WORLD





Rocket fuel pours from mixer at Thiokol's Huntsville, Alabama plant. For purity, cast Nickel-chromium-molybdenum blades are used to disperse oxidizer through the fuel.

Unique Stainless Steel Mixer Blades Boost Solid Rocket Power Production

Cleanliness is a must in sensitive and highly critical manufacture of solid rocket propellants.

Take mixing. Following reduction to precise size, particles of the inorganic oxidizer must be thoroughly dispersed through the viscous polysulphide. This must be done under conditions which exclude all contaminants, even trace metal from the equipment itself.

For efficiency. Thiokol Chemical Corporation uses a Baker Perkins Universal mixer specially equipped with "S"-shaped blades.

For product purity the blades are made of Type CF-8M (ACI designation) stainless steel. This is a 19% chromium, 10% Nickel, 2.5% molybdenum material with low (0.08%) carbon content. The alloy is highly resistant to corrosion by constituents of solid fuel, and by alkaline cleansers. Its use prevents contamination of the product by corrosion products and assures long blade contour life and maximum mixing efficiency.

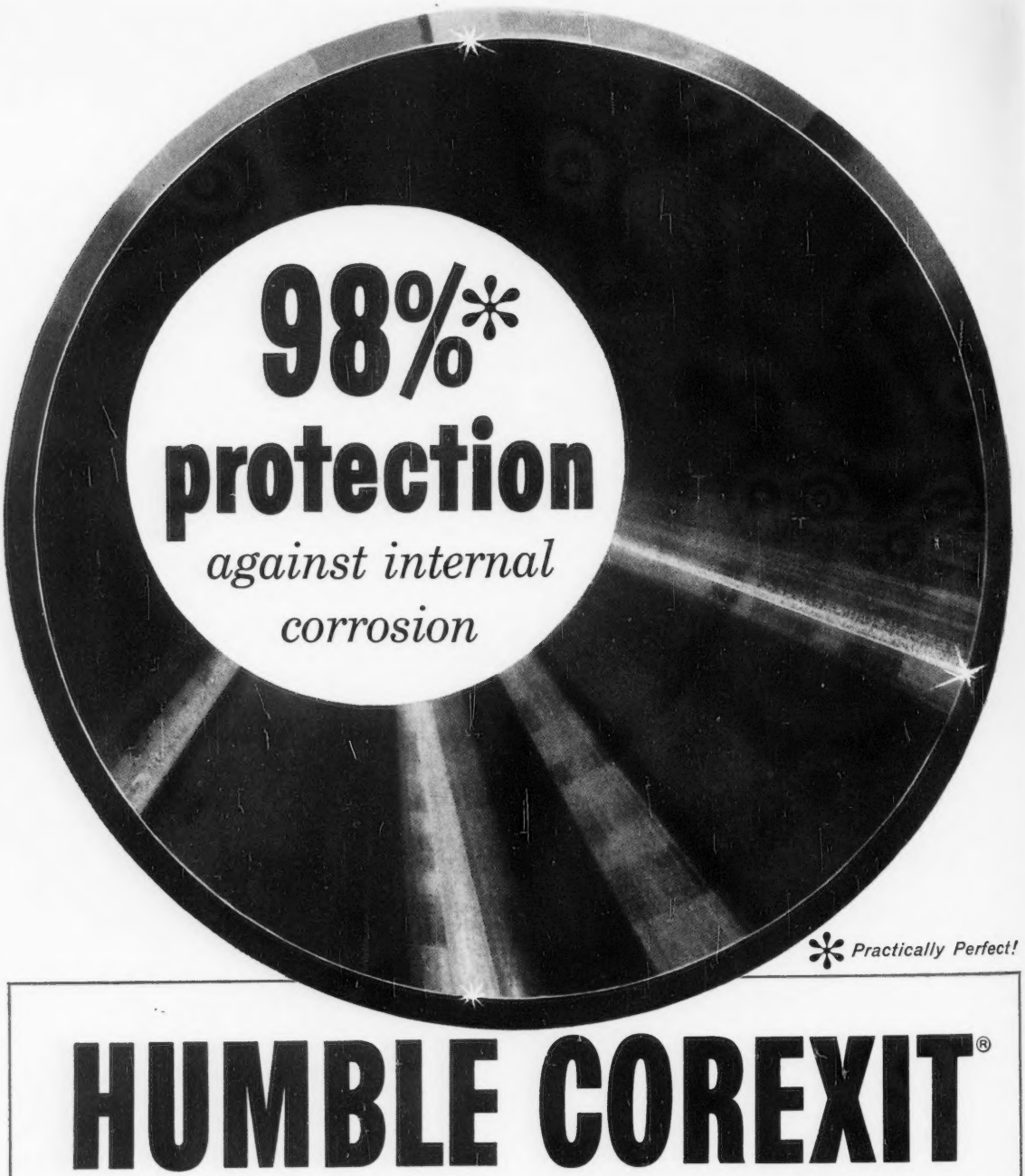
For economy the intricately-shaped blades are cast. Casting also permits smooth contours. Sharp corners and pockets that might trap contaminants or establish focal points for corrosion are eliminated. What's more, quick flush cleaning can be done reliably.

There may well be a place in your process equipment where stainless steel should be specified; for efficiency, for product purity, for economy. For an idea of what you can expect from these materials, write for a copy of the new Inco booklet, "Heat Resistant Castings, Corrosion Resistant Castings, Their Engineering Properties and Applications."

THE INTERNATIONAL NICKEL COMPANY, INC.
67 Wall Street INCO New York 5, N.Y.

INCO NICKEL

NICKEL MAKES STEELS PERFORM BETTER LONGER



HUMBLE COREXIT®

Save time and money previously lost to internal corrosion: use Humble COREXIT . . . COREXIT effectively and economically prevents internal corrosion in pipe still overhead, distillation towers, vapor recovery systems and other refining units. COREXIT will reduce or completely eliminate plugging or fouling. This protection against sludge, scale and corrosion cuts non-productive downtime to a minimum. For complete information on technical services and products, contact your Humble salesman.

HUMBLE

HUMBLE OIL & REFINING COMPANY

Equipment Services NEW PRODUCTS Materials Literature

Adhesives

Bonding of felt, wood, cork, insulation, fabrics, leather and some synthetic rubbers to themselves and to metals is claimed for Rex-N-Glue 244, an oil resistant synthetic rubber base adhesive made by Schwartz Chemical Co., Inc., 50-01 Second St., Long Island City 1, N. Y. The glue also is designed to be resistant to heat, cold, water and gasoline.

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A chemically resistant adhesive that is claimed to bond to most any type surface has been developed by Carboline Company, 32 Hanley Ind. Ct., St. Louis 17, Mo. Designed to be flexible and waterproof, Neoprene Adhesive F-1 is contact bonding: when two coated pieces are joined, there is an immediate "grab." No clamps or pressure is needed. This non-vulcanizing adhesive does not require a catalyst or accelerator, bonds to metals, concrete, brick, wood, rubber, glass, paper, canvas, cloth and all plastic materials except vinyls and polyethylenes, according to Carboline.

Coatings, Organic

Application of protective pressure-sensitive tape to large flat surfaces has been accomplished by the Westoak sheet coater, manufactured by Westoak Machine Corp., Oakland, Cal. The coater is used to apply tape to aluminum sheet, 84 inches wide and 200 inches long.

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Selection of coatings to fit individual requirements is explained in a bulletin on Carbomastic epoxy-tar protective coatings available from Carboline Company, 32 Hanley Ind. Ct., St. Louis 17, Mo. One section of the bulletin lists specific corrosion problems in industries such as chemical processing, pulp and paper, petroleum, etc., with recommended coating system given for the specific corrosion problem of each industry.

Fasteners

Non-deforming, heat and corrosion resistant split-head collar inserts with threads that are designed not to seize or gall are being manufactured by C.S.M. Screw Products Co., 2105 Southport Ave., Chicago 14, Ill. Made of stainless steel, the inserts are used for mounting non-slip plastic handles on electrical appliances. The insert is molded into the plastic.

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Rivets that can be applied with just a hammer are described in a brochure available from the Eldridge Co., 1608 Summer St., Philadelphia 3, Pa. Manufactured by the Southco Division of South Chester Corporation, a Drive Rivet kit is available which contains a variety of rivet grip lengths suitable for fastening materials of most common thicknesses. Rivets are placed in a drilled hole and struck with a hammer from the head side. There is no need to back up sheets or to reach the other side of the work, according to the manufacturer. Sheets are pulled together by expanding action of the rivet. These rivets also can be used in wood, expanding within the wood so that nothing protrudes to mar the reverse side.

Inhibitors

Lowered corrosion rates for steel and copper in recirculating cooling water systems are possible by use of a corrosion inhibiting compound, according to Bulletin CSP-935, available from Calgon Company, Hagan Center, Pittsburgh 30, Pa. Recommended treatment is with Hagatreat Formula No. 168, a chemical that is designed to provide corrosion control, to eliminate sludge deposition, to permit more concentration of make-up water and to simplify control procedures. Corrosion rates of less than one mil per year for steel and a few tenths of a mil per year for copper claimed for the inhibitor.

•

An oil soluble, non-metallic corrosion inhibitor designed to have no adverse effect on surfaces of lead bearings is described in a bulletin available from Atlas Powder Company, Wilmington 99, Del. Called Atpet 80, it is recommended for inhibition of corrosion on iron and steel; it has no adverse effect on lead surfaces when used at concentrations of 2 to 5 percent, according to Atlas. It was developed specifically to meet revised military specifications for preservative aircraft engine oils. The Air Force revised these specifications when it was discovered that oils were causing corrosion of lead-faced bearings in military aircraft engines.

•

Corrosion inhibitor for use in recirculating cooling water systems where reducing agents such as hydrogen sulfide may be present is discussed in Wrico 98 Bulletin, available from Wright Chemical Corp., 627 West Lake St., Chicago 6, Ill. Because it does not contain chromium or toxic materials, Wrico 98 is recommended for areas where disposal of such substances is a problem. Recommended treatment levels vary from 30 to 35 ppm for sweet waters and from 40 to 45 ppm for badly contaminated systems. The high level treatment of 40 to 45 ppm is the most required for severely contaminated systems, according to Wright Chemical.

•

Application methods for use of inhibitors to oil and gas well systems are described in a brochure on Kontol Corrosion Inhibitors, available from the manufacturer, Tretolite Company, Division of Petrolite Corp., 369 Marshall Ave., St. Louis 19, Mo.

Instruments

Ultrasonic flaw detection by use of a portable instrument is the subject of Bulletin T200, available from Branson Instruments, Inc., 40 Brown House Road, Stamford, Conn. The booklet includes operation, techniques and complete list of specifications for the Sonoray 5 flaw detector. By sending short pulses of acoustic energy at ultrasonic frequencies into metals, glass or plastics, the instrument indicates defects by variations in the object's characteristic trace on a cathode ray tube. Approximate depth of the flaw is determined by a pyramid marker superimposed on the base line.

•

Sensitivity range from 1 to 500 microns with data provided within minutes are features claimed for an automatic particle analyzer developed by Evans Research and Development Corp., 250 East 43rd St., New York 17, N. Y. Theory of the analyzer is based on a beta-ray, back scattering technique which measures magnitude of fluctuations or the "spread" in a random sampling operation under specific geometrical conditions. Its source of radiation is Carbon 14. Possible applications are in plastics, paints, drugs, foods, cosmetics, cements, insecticides, electronics, rubber, explosives, fuels, soils, fertilizers and ores.

Mergers

Acquisition of Acieries & Ateliers de Construction de Marpent, French producer of steel, railway cars and components and a variety of other industrial products has been announced by H. K. Porter Company, Inc., Porter Bldg., Pittsburgh 19, Pa.

•

Good-All Electric Mfg. Co., 112 West First St., Ogallala, Neb., has been purchased by Thompson Ramo Wooldridge Inc., of California.

Miscellaneous

Properties and applications of an improved copper-nickel-iron ductile permanent magnet alloy of the generic "Cunife" type are given in a brochure available from Hoskins Mfg. Co., 4445 Lawton Ave., Detroit 8, Mich. Properties listed include high coercive force and energy product values said to be comparable to those of cast or sintered Alnico 2 and 2, plus excellent ductility, malleability and machinability.

•

Industrial use of isotopes for radiographing all kinds of products is described in a booklet titled "Industrial Radiography With Radioisotopes" available from Picker X-Ray Corporation, 25 South Broadway, White Plains, N. Y. Booklet discusses advantages peculiar to isotopes in radiography, selection of proper isotope for a particular job and methods of making radiographic exposures.

•

Control of algae growths which can clog filters and injection formations by use of Nalco Cuprose is recommended in a technical data sheet available from Visco Products, 1020 Holcombe Blvd., Houston 25, Texas. This biological can be used in spray ponds, cooling systems, swimming pools, ponds, lakes and municipal reservoirs. Because strong solutions of Nalco Cuprose are corrosive to most metals, equipment of wood, rubber

(Continued on Page 44)

NEW PRODUCTS

(Continued From Page 43)

lined or ceramics should be used when continuous or proportional solution feed is required.

•

A marking ink that is non-corrosive to metals and that does not etch metals and most plastics but is removable by water has been developed by Organic Products Company, Box 428, Irving, Texas. It can be applied by rubber stamps and ordinary ink pads or by felt tipped pens.

•

A new waterproof sealant called Thoro-span for interior and exterior joints, glazing and calking is described in a brochure offered by Standard Dry Wall Products, Inc., New Eagle, Pa. Brochure No. 53 shows how the sealant is designed to provide permanent bond and seal on 23 types of building materials including wood, glass, aluminum, masonry and steel.

Plastics

Flexible magnetic stripping is being used on levels for electricians and metal workers so that the levels cling to metal surfaces, freeing the worker's hands for fitting and leveling. A magnetized vinyl plastic strip developed by B. F. Goodrich Industrial Products Co., Akron, Ohio, is used. This stripping also has been used to seal refrigerator doors and storm and screen doors to help close the openings.

•

Bearing oxidation is being prevented by use of a polyester film package. Small bearings manufactured by Miniature Precision Bearings, Inc., Keene, N. H., are sealed individually between two strips of 2½-mil Scotchpak polyester manufactured by Minnesota Mining and Manufacturing Co., 900 Bush Ave., St. Paul 6, Minn. The film can be heat-sealed at 300 to 400 F, is dimensionally stable and flexible from 70 to 240 F, has tensile strength to 10,000 psi and has burst strength from 32 to 50 psi.

•

Resistance to abrasion, plastic flow, cut-through and heat is claimed for a wire insulation made from a new fluorocarbon resin developed by W. L. Gore & Associates, Inc., 487 Paper Mill Road, Newark, Del. The new insulation basically consists of adding mineral-fiber particles to fluorocarbon resins. Improved resistance to abrasion and cut-through results from orienting over 90 percent of the fibers parallel to the wire surface. Random orientation would average about 50 percent, according to the developer.

•

A clear polyethylene film that is resistant to sunlight and that has an outdoor life three to four times that of standard, clear polyethylene film has been developed by E. I. du Pont de Nemours & Co., Wilmington, Del. Designated SR-300, the film will be available in four-mil gage in various widths.

•

Liquid, modified and stabilized sugar esters and derivatives for use as insolubilizer-plasticizer with polyvinyl alcohol and polyvinyl acetate have been developed by Holloway Sucro-Chemicals Corp., 57-02 48th St., Maspeth 78, Queens, N. Y. Called Hollesters, these products are designed to insolubilize polyvinyl alcohol in such compounds as emulsion type paints, coatings and polyvinyl alcohol films.

•

Chlorinated polether sheet stock made specifically for vessel lining applications is being marketed by United States Gasket Company, Plastics Division of Garlock Inc., 602 N. 10th St., Camden 1, N. J. The compound being used is Penton, a product of Hercules Powder Co., Wilmington 99, Del., which has resistance to corrosive, bleaching agents, solvents and plating solutions and is serviceable to 280 F. The material can be used to line vessels of unlimited size and is not subject to thermal shock, according to Garlock.

•

Use of tetrafluoroethylene resins in food handling service has been accepted by the Food and Drug Administration, according to E. I. du Pont, manufacturer of Teflon fluorocarbon resins. These resins are used in the extrusion of tubing, overbraided hose and pipe linings and to impregnate fibers and fabrics for such items as conveyor belts.

•

A portable repair kit designed for simple, economic and rapid permanent repairs to pipe, tubing, pipe joints and fittings, tanks and other vessels has been produced by Dresser Manufacturing Division, Bradford, Pa. Designated as the Util-Seal Kit, this epoxy resin repair kit contains proportioned materials for regular maintenance and emergency repairs. It contains individual portions of resin,

(Continued on Page 46)

"serving all pipelines"

N W E S

THE S. D. DAY COMPANY

MANUFACTURER'S REPRESENTATIVE FOR:

PITTSBURGH COKE & CHEMICAL CO.

Pitt Chem Hot Applied Enamels

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THE RUBEROID COMPANY

Inner and Outer Glass Pipe Wrap

Pipe Line Felts • Rock Shield

Distributor in the Southwest for:

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(Manufactured by American Coating Supply Co.)

MANUFACTURERS OF:

FORMULA 291

Skin Saver

*Fieldjointer Patent # 2763047

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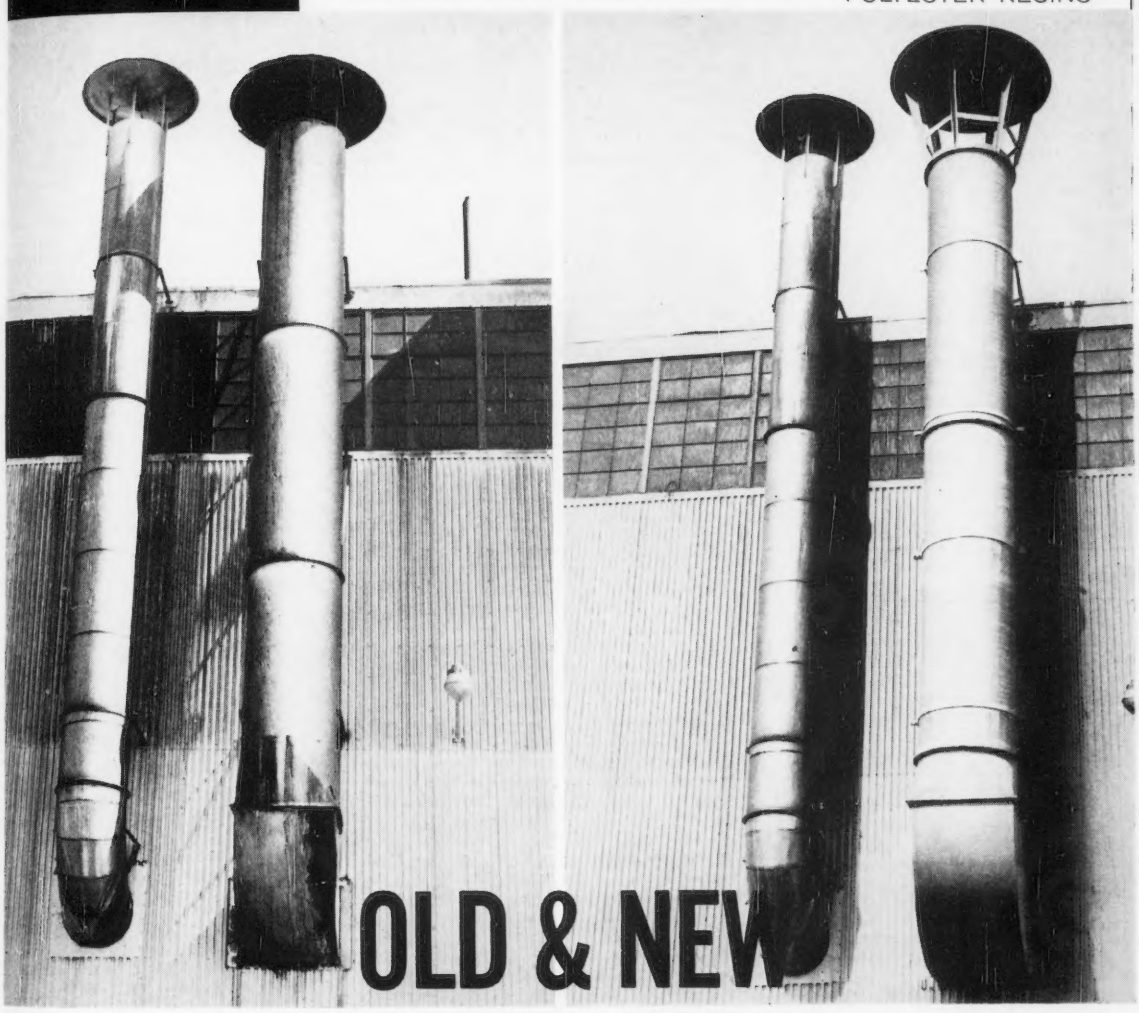
CYANAMID

CORROSIONEERING WITH LAMINAC®

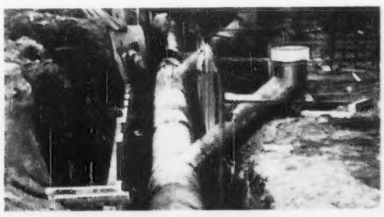
POLYESTER RESINS



in ventilating ducts for waste disposal

That stainless steel ventilating duct at the left was corroded in *only six months*, removing sulfuric acid fumes generated by an automatic aluminum anodizing machine. To solve this problem, the Ceilcote Company of Cleveland, Ohio, designed and fabricated a maintenance-free stack utilizing Laminac® polyester resin reinforced with glass mat. The initial cost of the new stack on the right was 30% *less* than steel. There was no sign of corrosion after *eighteen months* of severe service – an example of how reinforced Laminac resists acids, gases and fumes – at low cost and with complete freedom from maintenance. Reinforced Laminac can solve *your* corrosion problems. For more information, or technical assistance, get in touch with any Cyanamid office listed below.

NEW CORROSION-PROOF PIPE, OF REINFORCED **LAMINAC**, PROVIDES LOW-COST ANSWER TO CORROSIVE WASTE



Laminac, reinforced with glass mat, is the *heart* of this reinforced plastic waste pipe made by Ceilcote. It is used in metal-working, chemical, textile and allied industries. This standard line of pipe now handles waste pickling liquor with 10-15% dilute sulphuric acid. Reinforced Laminac features high strength with low weight, positive sealing, lower installed cost and lasting corrosion resistance.

CYANAMID AMERICAN CYANAMID COMPANY • PLASTICS AND RESINS DIVISION

SOUTH CHERRY STREET, WALLINGFORD, CONN. *OFFICES IN:* BOSTON • CHARLOTTE • CHICAGO • CINCINNATI • CLEVELAND • DALLAS • DETROIT • LOS ANGELES MINNEAPOLIS • OAKLAND • PHILADELPHIA • ST. LOUIS • SEATTLE • WALLINGFORD, CONN. *IN CANADA:* CYANAMID OF CANADA LIMITED, MONTREAL • TORONTO

NEW PRODUCTS

(Continued From Page 44)

activator and fibrous reinforcer with a collector ring and pressure relief vents for use during the setting-up period.

•

A new vinyl tubing designed for use with pneumatic tools such as spray painting equipment has been developed by the DeVilbiss Company, Toledo, Ohio. The hose is said to be suitable also for use in air systems laden with oil. The company also has available its Synflex Nylon tubing with solvent resisting qualities for use in transmitting fluids to spray equipment. This flexible, lightweight, translucent hose will not affect paint colors, according to DeVilbiss.

•

Molded sheets and surface coatings of chlorotrifluoroethylene are being used to protect industrial gages and instruments. Gages produced by Jerguson Gage and Valve Co., Burlington, Mass., are protected with this coating for resistance to handling acids and other corrosive fluids. This polymer is used to provide a transparent and highly resistant surface for glass windows in gages.

Pumps

High pressure, stainless steel pumps to handle most processing liquids are available from Sonic Engineering Corp., 146 Selleck St., Stamford, Conn. The pumps are made in two capacities from 1 to 30 pgm with pressures to 400 psi.

•

A centrifugal pump with canned stator and flow-through type rotor-impeller is described in Bulletin M-60 available from the Corley Co., Inc., 685 Myrtle Ave., Boonton, N. J. The pump and motor are combined in one unit to eliminate drive shafts, couplings, packing glands and bases. Handling capacities to 115 gpm against heads to 31 feet, these pumps are designed for corrosive, toxic, flammable, noxious, radioactive and volatile liquids. Installed in pipelines in sizes to two-inch diameters, they require only biannual inspection of bearings, according to the manufacturer. This inspection can be done with a screwdriver and does not require disturbing the pipeline.

Make SEA WATER
-ANYWHERE!
SEA RITE SALT
for CORROSION STUDIES
4½ oz. Sea Rite Salt crystals in one gallon tap water produces laboratory sea water quickly, inexpensively. Provides uniform, constant results based on 77 composite analysis. Immediate delivery — any quantity. Technical literature on request. Write, wire, phone now!
LAKE PRODUCTS CO., Inc.
ST. LOUIS 25, MISSOURI

Welding

Cold bonding, threadless stitching, sealing or welding of plastic films and synthetic woven or non-woven fabrics has been developed by Ultrasonic Industries, Inc., 141 Albertson Ave., Albertson, Long Island, N. Y. The new technique uses high frequency sound waves above the human audibility range to make splices or seals. No heat or dielectric currents are used, thus eliminating distortion or chemical change in the material being processed.

•

Brazing techniques on all commercial brazeable metals and alloys are described and illustrated in a manual offered by All-State Welding Alloys Company, Inc., 249-55 Ferris Ave., White Plains, N. Y. Details are given on such basic brazing functions as heat, flame and torch settings, alloy selection and joint design. Common brazing problems are discussed in sections on copper, copper alloys, aluminum, cast iron, steel and stainless steel brazing.

MEN in the NEWS

Fred Kutch has been appointed sales manager for petroleum and industrial products for Neptune Meter Company's Liquid Meter Division, 19 West 50th St., New York 20, N. Y.

•

NACE member **Fred M. McConnell** is associated now with Spence & Sullivan, Inc., Torrance, Cal.

•

G. W. Prager has been appointed sales representative in the states of Colorado, Wyoming, Montana, Utah and Idaho for Standard Pipeprotection Inc., 3000 South Brentwood Blvd., St. Louis 17, Mo.

•

Marvin Reichle, an NACE member, has been appointed assistant sales manager for Plastic Applicators, 7020 Katy Rd., Houston, Texas.

•

Two young executives of Union Carbide Plastics Company have been named Alfred P. Sloan Fellows in executive development at Massachusetts Institute of Technology. They are **Joseph F. Rocky,** assistant director of development at the company's Bound Brook, N. J., Development Laboratories, and **Ernest F. Turner,** area supervisor of polystyrene at the company's Marietta, Ohio, plant.

•

A. Henry Schutte has been appointed to the senior staff of the chemical engineering section at Arthur D. Little, Inc., Cambridge 40, Mass.

•

Roland H. Wilson has been appointed works manager at the Riverside, N. J., plant of Riverside-Alloy Metal Division, H. K. Porter Company, Inc.

•

Robert S. Thompsen has been appointed to the newly created position of manager of technical activities for the Porcelain Enamel Institute, 1145 19th St., N. W., Washington 6, D. C.

•

Cloyd A. Snavely has joined Sifco Metachemical, Inc., 935 E. 63rd St., Cleveland, Ohio, as general manager.

A. H. Sonnenschein has been appointed director of planning at Polarad Electronics Corp., 43-20 34th St., Long Island City, N. Y.

•

Two West Coast sales engineers have been appointed by the William Powell Co., Cincinnati, Ohio: NACE member **William P. Simmons** as regional sales engineer for the San Francisco area and **Robert A. Miles** as district sales representative in Los Angeles.

•

Earl E. Hall has been appointed sales manager of Colvin Laboratories, Inc., 364 Glenwood Ave., East Orange, N. J.

•

W. J. Ferracone has been named general sales manager of Miles Chemical Company, 1127 Myrtle St., Elkhart, Ind.

•

Gerald Lewis has been appointed director of product development for Cooper Alloy Corporation, Hillside, N. J.

•

A. Arthur Kwapisz has been named plant manager of Pittsburgh Chemical Company's new carbon plant under construction at Catlettsburg, Ky.

•

K. Russell Knoblauch has been appointed market sales manager for Minneapolis-Honeywell Regulator Company's Industrial Products Group in Philadelphia, Pa.

•

Leo O. Witzenburgh of Cleveland Worm & Gear Division of Eaton Manufacturing Company was elected president of the American Society of Lubrication Engineers.

•

John L. Young has been appointed vice president of engineering technology for U. S. Steel Corporation, 2306 Gulf Bldg., Houston 2, Texas.

•

Omer Rhodes has joined the staff of Clemtex, Ltd., 248 McCarty Drive, Houston, Texas.

•

Presson S. Shane was elected vice president of Atlantic Research Corporation, Alexandria, Va.

•

Joseph W. Selden has been elected divisional vice president for the chemical division of Minnesota Mining and Manufacturing Co., St. Paul 6, Minn.

•

Gilbert C. Strubell has been appointed administrative director of metallurgy and research for American Brass Company's Torrington Division.

•

Arthur F. Becker was named vice president of manufacturing for General Rubber Corp., Tenafly, N. J. **Robert L. Swenson** was named vice president in charge of engineering.

•

NACE member **R. L. von Hohenleiten** has been named manager of anode sales for National Carbon Company, division of Union Carbide Corp., 100 E. 42nd St., New York 17, N. Y.

•

Leland E. Wells has been promoted to vice president of engineering for the Exide Industrial Division of the Electric Storage Battery Co., Philadelphia, Pa.

•

Kenneth T. Wood has been named ceramics coordinator for the New Products Division of Corning Glass Works, Corning, N. Y.

Program Information Given on Five Corrosion Short Courses

Program information and dates are given on five of the ten corrosion control short courses held in cooperation with NACE. Program information is given below on short courses to be held at Washington University, University of California, University of Oklahoma, Ohio State University and the Florida General Conference Short Course.

Other short courses held in cooperation with NACE are the Appalachian Underground Corrosion Short Course at the University of West Virginia to be held June 6-8, 1961, University of Illinois' biannual course, and three short courses sponsored annually by the Shreveport, Houston and Sabine-Neches Sections.

Ohio State University September 12-16

A short course on process industry corrosion will be held September 12-16 at Ohio State University, Columbus. It will be presented by the University's Department of Metallurgical Engineering in cooperation with NACE's Technical Group Committee T-5 on Corrosion Problems in the Process Industries.

Enrollment will be limited to 150. Registrations accompanied by full payment of fee ($65) should be made as early as possible and by August 26 at the latest. Checks should be made payable to Ohio State University.

Registrations should be mailed to M. G. Fontana, chairman, Department of Metallurgical Engineering, Ohio State University, 140 West 19th Ave., Columbus 10, Ohio.

A complete schedule of lectures was published on Page 55 of the May issue of CORROSION.

University of Oklahoma September 28-30

A series of technical papers, field demonstrations and panel discussions on corrosion control will be offered during the September 28-30 corrosion control short course offered by the University of Oklahoma's College of Engineering and Extension Division in cooperation with NACE's Central Oklahoma Section.

Speakers and program have been selected to better acquaint engineers and operating personnel with the practical aspects of corrosion control. Sessions will be given on corrosion fundamentals and on corrosion problems in oil production, pipelines, public utilities, plants and refineries. Instrumentation for corrosion surveys and mitigation will be demonstrated and discussed. A special feature of the program will be a field demonstration of corrosion measurement techniques and corrosion control in buried structures.

Sessions will be held at the Extension Study Center of the University of Oklahoma at Norman. Registration forms and additional information can be obtained from M. L. Powers, Director, Business and Industrial Services, Extension Division, University of Oklahoma, Norman, Okla.

A complete listing of program topics and speakers for the course are scheduled for publication in the September issue of CORROSION.

University of California October 3-5

A short course in corrosion will be offered October 3-5 at the University of California in San Francisco in conjunction with the 1960 Western Region Conference set for October 6-7, also in San Francisco.

Jointly sponsored by NACE and the University of California, Berkeley, and the University of California, Los Angeles, the short course includes sessions on a variety of topics ranging from fundamentals of corrosion to specialized corrosion problems such as corrosion in salt water, sulfuric acid, process water, liquid metals and in such equipment as heat exchangers and pumps.

A detailed schedule of topics and speakers for the course are given below.

Registration information can be obtained from I. Cornet, University of California, Engineering and Sciences Extension, 2451 Bancroft Way, Berkeley 4, Cal.

October 3

8 am Registration.
8:20 am Introduction and welcome.
8:30 am Fundamentals of Corrosion: physico-chemical, electrochemical, metallurgical aspects.
10:30 am Solid State Theory in Corrosion: Fundamentals, by W. E. Tragert, general Electric Co., Schenectady, N. Y.
1:30 pm Solid State Theory in Corrosion: Oxidation, et al. by W. E. Tragert.
3 pm Corrosion in High Purity and Reactor Water, by J. E. Draley, Argonne National Laboratory, Argonne, Ill.
4:15 pm Reactor Materials: Stainless Steel, Aluminum, Zirconium, by W. E. Berry, Battelle Memorial Institute, Columbus, Ohio.

October 4

8 am Corrosion in Process Water, by F. L. Whitney, Jr., Monsanto Chemical Co., St. Louis, Mo.
10 am Corrosion in Salt Water, by T. P. May, International Nickel Co., Inc., Wrightsville Beach, N. C.
1:30 pm Process Chemicals: Sulfuric Acid, by G. Nelson, Shell Development Co., Emeryville, Cal.
2:30 pm Process Chemicals: Sodium Hydroxide, Chlorine, Hydrochloric Acid, by R. C. Vasey, Dow Chemical Co., Pittsburgh, Cal.
2:30 pm Corrosion in Liquid Metals, by D. E. McKenzie, Atomics International, Canoga Park, Cal.

October 5

8 am Heat Exchangers, by A. German, Scovill Manufacturing Co., New York, N. Y.
10 am Pumps, by V. S. Lobanoff, Ingersoll-Rand Co., New York, N. Y.
1:30 pm Welding.
2:30 pm Non-Metallic Linings, by M. K. Weber, Shell Development Co., Emeryville, Cal.
3:30 pm Inspection Problems, by E. H. Tandy, Standard Oil Company of California, El Segundo, Cal.

Florida General Conference November 7-11

The annual Florida General Conference Corrosion Short Course, scheduled for November 7-11, is sponsored by the Miami and Jacksonville NACE Sections. It will be held at the Key Biscayne Hotel in Miami.

Tentative schedule for the short course was published on Page 68 of the July issue of CORROSION.

Washington University June 5-9, 1961

Tentative program listed below for the June 5-9 Corrosion Short Course to be co-sponsored by NACE's Greater St. Louis Section and Washington University shows an emphasis on the fundamental approach to corrosion studies.

The course will begin with fundamental studies of corrosion theory and mechanisms, testing, then progress to study of control and preventive methods through cathodic protection, inhibitors, material selection, plastics and protective coatings.

Monday, June 5

1. Corrosion theory and mechanism, types of corrosion.
2. Corrosion testing: (a) fundamental techniques and their relation to practice, (b) empirical techniques (laboratory testing with attention on duplicating field conditions; field testing and evaluation).

Tuesday, June 6

1. Corrosion control and preventive methods: anodic passivation and cathodic protection.
2. Theory and application of inhibitors.
3. Process control and change.

Wednesday, June 7

1. Corrosion resistance and application of the newer metals: titanium and zirconium and their alloys.
2. Corrosion resistance and application of steel and low alloy steels.
3. Corrosion resistance and application of copper and copper base alloys.
4. Corrosion resistance and application of the light metals.

Thursday, June 8

1. Ferritic and martensitic stainless steels.
2. Austenitic stainless steels.
3. Nickel and nickel base alloys.
4. Special alloys.

Friday, June 9

1. Rigid plastics.
2. Plastic and elastomeric linings.
3. Organic coatings.
4. Metallic and metallo-organic coatings, including refractories, plasma-jet, etc.

Northeast Region

Time Schedule and Abstracts Are Given For Oct. 11-14 Huntington Conference

Schedule of events for the October 11-14 Northeast Region Conference to be held at Huntington, W. Va., is given below. Conference meetings will be held at the Hotel Prichard.

Abstracts for some of the 33 technical papers to be presented in eight symposia also are given below under symposia headings. These include program changes made since the material was published on Page 72 of the July issue of CORROSION.

Tuesday, October 11

12:30-6 pm Registration
1-5 pm Technical Practices Committee meetings (T-6A, T-6A-12, T-7A and T-5A)
6 pm Northeast Region Trustees Dinner Meeting

Wednesday, October 12

9 am-5 pm Registration
9-12 noon Protective Coatings for Atmospheric Exposure Symposium
9-12 noon Inhibitors Symposium
9-12 noon Technical Practices Committee Meetings (T-5E, T-2K and T-3G-1)
11-6 Plant Tour (Kaiser Aluminum)
1-5 pm Plant Tours
1-5 pm Technical Committee Meeting (T-6F)
7:30 pm Special Event: Fun and Eats, Mountaineer Style

Thursday, October 13

9-5 Registration
9-12 noon Protective Coatings for Immersion Service Symposium
9-12 noon High Temperature Corrosion Symposium
2-5 pm Fresh Water Corrosion Symposium
2-5 pm Plastics Symposium
6:30 pm Fellowship Hour
7:30 Banquet

Friday, October 14

9-12 noon Underground Corrosion Symposium
9-12 noon Chemical and Refinery Industry Process Corrosion Symposium

•

Wilmington Section had Harvey E. Atkinson of E. I. du Pont de Nemours & Company speak on the use of thermoplastics for chemical process equipment at the May 25 meeting.

POSITIONS WANTED AND AVAILABLE

Active and Junior NACE members and companies seeking salaried employees may run without charge two consecutive advertisements annually under this heading, not over 35 words set in 8 point type. Advertisements to other specifications will be charged for at $10 a column inch.

Positions Available

Corrosion Engineer—To head up and expand gas distribution corrosion control program. Please address resume of past experience, education and personal data to J. L. McAdam, Illinois Power Company, 500 S. 27th Street, Decatur, Ill.

Corrosion Engineer with 10 years of diversified chemical processing problem solving experience is required by a large multi-plant chemical organization. Will evaluate current methods and procedures and set specifications for new construction. Write Box 60—24, CORROSION.

Positions Wanted

Petroleum-Corrosion Engineer, B.S. 1952, experience in cathodic protection and oil field operations. Desire position, with growth potential, with oil or gas company. Will relocate. Age 35, married. Resume on request. CORROSION, Box 60—23.

Engineer—Nine years' experience electrochemistry and Diesel, gasoline engines with corrosion emphasis. Other experience research and development, heavy in instrumentation. Want full-time corrosion position. Age 39. BS physics. CORROSION, Box 60-25.

Corrosion Engineer—Seven years' corrosion control and three years' diversified experience with both gas distribution and pipeline companies. BS in Ch.E. Married, will relocate. Resume on request. CORROSION, Box 60-26.

Positions Available

DISTRIBUTORS WANTED

50-year-old rubber manufacturer seeks distributors now calling on pipe line industry. Product is pipe wrap putty tape which is used to improve effectiveness of regular plastic overwrap tapes. Product enjoying excellent repeat sales in areas where introduced and we are now looking for additional distribution.

WEST AMERICAN RUBBER COMPANY
410 North Avenue 19
Los Angeles 31, California

WANTED: Graduate chemist or chemical engineer for product development of industrial maintenance coatings.

EXPERIENCE: Up to five years general industrial maintenance coatings formulation development.

DUTIES: Development and applications research of chemical and corrosion resistant coatings, epoxy plastics for structural applications and finishing systems for building maintenance. Must be able to meet and discuss coatings problems with sales personnel and customers and have supervisory ability or potential.

SALARY: Commensurate with experience, excellent benefits.

LOCATION: Truscon Laboratories, Inc. Division, Detroit, Michigan.

In reply, please indicate availability for an interview in Detroit, salary requirement, education and experience. Forward all replies in confidence to C. M. Jackson, Devoe & Raynolds Company, Inc., P.O. Box 328 Louisville 1, Ky.

Northeast Region Conference ABSTRACTS

FRESH WATER CORROSION

Once-Through Cooling Systems, by P. R. Puckorius, Nalco Chemical Co., Chicago, Ill.

Materials for Construction for Natural Fresh Waters, by G. B. Elder, Union Carbide Chemicals Co., South Charleston, W. Va.

Discusses behavior of common metallic and nonmetallic materials of construction in natural fresh waters. Types of corrosion to which each material is susceptible is covered and anti-corrosion measures reviewed.

UNDERGROUND CORROSION

Plastic Pressure-Sensitive Tapes as Pipe Coating Materials, by George F. Roberts, Seamless Rubber Co., New Haven, Conn.

Considers history of plastic pressure-sensitive tapes and new types, including high density polyethylene blends. Describes usage and performance of these materials.

Cathodic Protection of Well Casings, by W. R. Lambert, United Fuel Gas Co., Charleston, W. Va.

Discusses cathodic protection principles as applied to well casing corrosion. Use of casing probes to determine extent of corrosion on interior is correlated with surface measurements of potential-to-ground of casing to determine practical means of testing for corrosion of the coating. Comparison is given of various protective coatings used on casing exterior under cathodic protection.

Maintenance Practices of Corrosion Control Facilities for Underground Structures, by D. A. Tefankjian, Texas Eastern Transmission Corp., Shreveport, La.

Discusses maintenance practices for corrosion prevention facilities for underground structures, showing actual operation of a program aimed toward corrosion preventive measures. Emphasis is placed on maintaining effective protection after an adequate system has been provided. Survey profiles and report forms to be shown and their importance discussed.

INHIBITORS

Corrosion Inhibitor Efficiency in Aqueous Ethylene Glycol Solutions, by J. C. Cessna, National Carbon Co., Parma, Ohio.

Reports on effect of inhibitors on corrosion of iron, aluminum and copper alloys in aqueous systems. Effect on pre-corroded surfaces and a correlation of weight loss with electrical resistance changes are discussed also.

Notes on Corrosion Inhibitors, by C. W. West, Union Carbide Chemicals Co., Ardsley, N. Y.

Reviews corrosion inhibitors with emphasis on application. Inhibitors for ferrous and non-ferrous metals are considered in neutral aqueous liquids, aqueous solutions of acids, aqueous solutions of bases, aqueous solutions of organic compounds and non-aqueous or organic media. Volatile inhibitors are discussed and reference made to use of environmental stabilizers such as antioxidants. Also considers problems in use of corrosion inhibitors.

PROTECTIVE COATINGS IMMERSION

Pre-Shrunk Films—Those Remarkable Epoxies Again, by Richard G. Fortener, Jones-Dabney Co., Louisville, Ky.

Gives basic consideration of factors affecting cure and performance of air-drying epoxy resin coatings. Compares advantages and disadvantages of 100 percent solids systems with solvent formulations, including performance results of the two. Future prospects are given for solvent-free system.

Tonic Solvents—What You Should Know About Them, by E. G. Meiter, Employers Mutual Insurance Co., Wausau, Wis.

Discusses hazards of toxic solvents in coating systems from standpoint of toxicity, dermatitis and fire and explosion. Explosive properties of solvents to be demonstrated in small explosion testing apparatus.

Practical Considerations Governing Successful Coating Applications, by R. E. Robinson, Hooker Chemical Co., Niagara Falls, N. Y.

New techniques, experimentation and a thorough investigation of cost factors are discussed to show an improved philosophy of coating work. Discusses practical aspects of obtaining the most for the coating dollar.

Penton Coatings for Industrial Use, by O. J. Britton, W. A. Carlson and J. R. Little, Pfaudler Company, Rochester, N. Y.

Describes use of Penton as coating material. Gives its properties, techniques of application as

a coating including recently developed water-based suspension system. Quality control tests, laboratory and field experience and effect of chemical attack on coating to be illustrated by slides.

HIGH TEMPERATURE

Behavior of Engineering Alloys in Hot Ammonia Atmospheres, by J. J. Moran and J. K. Mihalisin, International Nickel Co., New York, N. Y.

High Temperature Furnace Corrosion, by R. D. Merrick, Esso Research and Engineering Co., Madison, N. J.

Review of Russian Literature on High Temperature Chlorine Conversion, by R. E. Gachenback, American Cyanamid Co., New York, N. Y.

High Temperature Sulfur Corrosion, by R. A. Miller, American Brakeshoe Co., Elyria, Ohio. Reviews high temperature corrosion of alloys exposed to sulfur-bearing gases under reducing and oxidizing conditions. Laboratory data and case histories of corrosion problem will be given.

A New Design for Reinforced Plastic Tanks, by F. H. Humphrey, Toronto, Canada, and W. R. Wandrop, Metal Cladding, Inc., North Tonawanda, N. Y. Describes development and advantages of reinforced polyester and epoxy plastic tank construction incorporating helical cable windings on exterior to relieve the plastic itself much of the stress of construction.

A Comparative Study of Corrosion Resistance of a Bisphenol Resin, an Isophthalic Resin and a General Purpose Polyester Resin, by A. F. Torres and S. S. Feuer, Atlas Powder Co., Wilmington, Del. Resistance of three types of polyester resins is given at 75 and 210 F in eight different acid and alkaline solutions as determined by changes in mechanical properties after exposure. Effect of various laminate construction methods on resistance was determined and found to have significant effect on stability of the resin system.

Plastics as Construction Materials in Industry, by J. Bigos, U. S. Steel Corp., Monroeville, Pa. Primary interest is in combining steel and plastic to produce a product superior to that offered by either material when used alone. Considers building materials of all types.

Design of Glass-Reinforced Plastic Structures, by Lawrence Fischer, Grumman Aircraft Engineering Corp., Bethpage, N. Y. Mathematical procedure is given for analyzing structural behavior of laminates constructed of individual layers of glass fabric or roving supported by a resin and subjected to loads in plane of the laminate. Discusses means of designing orientation of each layer so that applied loads are resisted most effectively. Also considers effects of material variables on the theory and proposed future work.

CHEMICAL & REFINERY INDUSTRY

Corrosion Characteristics and Uses of Glassed Steel, by A. S. Levy, Pfaudler Co., Rochester, N. Y. Contrasts corrosion behavior of glassed steel with metallic corrosion. Problems concerning galvanic attack, concentration cells, chlorides and electrochemical reactions are discussed in relation to glassed steel. Describes chemical and temperature limitations and uses of glassed steel. Examples of glassed steel construction in pharmaceutical and chemical industries are given.

Corrosion of Weak Acids Under Heat Transfer Conditions, by N. D. Groves, C. M. Eisenbrown and L. R. Scharfstein, Carpenter Steel Corp., Reading, Pa. Describes corrosion of various type alloys as influenced by heat-flux through the metal. Gives data on corrosion of alloys in phosphoric, formic, and acetic acid solutions at three different concentrations and at various temperatures, including those above the boiling point of a liquid.

Chloride Corrosion and Fouling in Catalytic Reformers and Naphtha Pretreaters, by E. B. Backensto and A. N. Yrick, Socony Mobile Oil Co., St. Louis, Mo. Discusses fouling problem along with corrosion noted. Describes laboratory studies of the problem and use of ammonia or amine-type inhibitors to suppress corrosion.

Fabrication vs Corrosion, by E. W. Kleefish, Nooter Corp., St. Louis, Mo. Discusses fabrication techniques and resultant effects on corrosion rates. Comparison of fabrication methods for various materials and design factors are described in relation to proper use of the materials. Quality control of materials as aid in corrosion prevention is discussed.

Corrosion Resistance of Steels, by S. W. Shepard, Chemical Construction Corp., New York, N. Y. Application of knowledge of corrosion principles and proper use of materials are discussed as means to reduce cost of corrosion in chemical process industry. Suggested means of achieving this goal are given.

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...This low cost fatty diamine makes high quality corrosion inhibitors!

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- It exhibits excellent stability in corrosive environments.
- It is soluble in hydrocarbons . . . as the free diamine or as its salts of high molecular weight acids.
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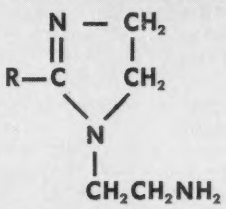

Nalcamine G-39M is a mixture of diamino imidazolines, consisting predominantly of the diamine shown above where the mixed alkyl chains are heptadecenyl and heptadecadienyl.

TYPICAL ANALYSIS

Apparent Molecular Wt.	422
Apparent Combining Wt.	211
% Imidazoline	94.0
% Titratables	99.2
Spec. Gravity at 60°F.	0.948
Pour Point °F.	25
Viscosity at 60°F. cps	334
Density—Lbs. per gallon	7.91
Color	Black

SHIPPING

Nalcamine G-39M is shipped in fifty-four gallon, bung, unlined, non-returnable steel drums. Weight of each drum is approximately 420 lbs.

HANDLING

Nalcamine G-39M can be handled or stored in iron or mild steel. It should be stored in a closed container.

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Nalco CHEMICALS

Wrought Iron plate cut by hand from fire curtain of Marina Yacht Harbor Pier, San Francisco, California—The dense and compact nature of Wrought Iron's protective scale is clearly evident. Its adherence is permanent: on plate after 26 years, and on tubular goods after 73 years of salt water service.

Steel tie rod after cleaning and measuring adjacent to deck where paint was still intact—Note chipped and spalled appearance of scale on rimmed steel tie rod. This tie rod was originally 1" material. When cleaned of rust, the rod measured 0.481", 0.490" and 0.697" on three different diameters.

73 years' exposure to salty sea water produced only 3 mils/year maximum pit depth on Wrought Iron

A structural inspection of the Marina Yacht Harbor Piers in San Francisco, Calif., revealed once again, the decided superiority of Wrought Iron over steel in salt water environment.

Maximum pit depth on Wrought Iron after 73 years' exposure to sea water and sea air was 210 mils: approximately 3 mils/year.

Steel similarly located, showed perforation of ¼" plate after 18 years: approximately 14 mils/year. Severance of one-inch steel rods occurred in 26 years.

No case of Wrought Iron perforation was evident. Either in ¼" plate over a period of 26 years, or in tubular goods over a period of 73 years.

Corrosion researchers reported the following detailed findings in their structural inspection:

- Scale on Wrought Iron was heavy and dense. Scale adhered so tightly that a diamond pattern hammer was required to remove it. Scale on steel was equally thick. But it was so friable that light hammering was sufficient to remove it.
- Some scabbing occurred on Wrought Iron at mean high tide (MHT). But, in every case where a scab was hammered off, a dry inactive pit was found.
- At MHT, the maximum pit depth of Wrought Iron was 210 mils. Steel at the same position showed depths of as much as 360 mils. This occurred even though the steel had been in service less than 26 years. Wrought Iron had already withstood 73 years' service.
- In the area of MHT, where the maximum Wrought Iron pit depth was 210 mils in 73 years, the ¼" (250 mils) steel plate had perforated in less than 18 years.
- Wrought Iron, ¼" thick, adjacent to the ¼" steel plate which had perforated in 18 years, showed a maximum loss of 187 mils in 26 years. This occurred in spite of the fact that the Wrought Iron had been subjected to extreme crevice corrosion not obtaining on the steel.
- One-inch steel tie rods passing through MHT showed reduction in diameter ranging from 293 mils to one inch. This would give pit depths (corrosion from one side only) of from 147 to 500 mils in 26 years. This compares with Wrought Iron pits of 210 mils in 73 years.
- No plausible arguments involving small amounts of copper and nickel can be used to invalidate these data. The compositions of the steel and Wrought Iron are very similar for these elements.

The most casual observation reveals use of two different materials in these docks. The Wrought Iron, after 73 years' exposure to salt water and salt air, is today in much better condition than the steel installed 26 years ago.

A complete inspection report of the Marina Yacht Harbor Piers is available on request. Write A. M. Byers Company, Clark Building, Pittsburgh 22, Pa.

BYERS 4-D WROUGHT IRON
TUBULAR AND FLAT ROLLED PRODUCTS
Corrosion costs you more than Wrought Iron

Heavy Industrial Region Center Site of 1961 Corrosion Show

The 1961 Corrosion Show to be held in conjunction with the 17th Annual NACE Conference at Buffalo, N. Y., March 13-17, will be at the center of one of North America's heaviest industrialized areas. Over half the U. S. population and over 70 percent of all Canadians live within 500 miles of Buffalo—a concentration of 96 million people.

Industrial Areas Represented

Exhibitors participating in the 1961 Corrosion Show can expect a large majority of registrants at the conference to be from this Northeast industrial area, according to R. W. Huff, Jr., NACE Exhibits Manager. About 45 percent of registrants at past NACE conferences and Corrosion Shows has come from the industrial areas of New York, Pennsylvania, New Jersey, Wisconsin, Michigan, Illinois, Indiana and Ohio. An even larger percentage of Buffalo Conference registrants will come from the chemical, steel and other industries of the area, Mr. Huff estimates.

The 1961 Corrosion Show will be held in the Hotel Statler-Hilton, on the same floor as the technical meetings of the conference, thus affording registrants a convenient opportunity to visit the displays, Mr. Huff adds.

No technical symposia meetings are scheduled for Tuesday afternoon, March 14, the opening date of the Corrosion Show, so that all registrants can attend the show.

Corrosion Show Brochures Mailed

Approximately 2000 copies of a brochure giving complete information on the 1961 Corrosion Show have been mailed to prospective exhibitors. These brochures include complete information on the show, floor plan of the exhibit area with booths indicated, prices for booths and forms to be completed by exhibiting companies.

A sample is included in the brochure of an invitation which can be obtained by exhibitors wishing to extend invitations to customers, prospects and friends.

Color Slide Presentation

Information also is given on a color slide presentation to be made for use by NACE regions and sections. The color slides will be photographed of displayed products at no cost to exhibitors. The slide presentation will include a narrative script describing the products on display so that persons not attending the Buffalo Conference will have an opportunity to see the diversity of corrosion control products, services, processes and materials exhibited at the 1961 Corrosion Show.

SECTION CALENDAR

August
2 Shreveport Section. Ladies Night
3 Teche Section. Petroleum Club
9 Houston Section. To be at Houston Engineers' Club.

September
7 Teche Section. Petroleum Club.
9 Birmingham Section.
12 Greater St. Louis Section. Non-Destructive Test Methods, by John Bobbin, Branson Instruments.
12 Kansas City Section.
12 Central Oklahoma Section.
12 North Texas Section. Torch Restaurant, Dallas, 6:30 pm.
13 San Francisco Bay Area Section.
13 Houston Section. Houston Engineers Club.
13 Permian Basin Section.
15 Detroit Section.
15 Vancouver Section.
20 Cleveland Section.
20 Alamo Section.
26 Tulsa Section.
27 Panhandle Section.
27 Southwestern Ohio Section.

South Central Region

Abstracts of Tulsa Papers Are Given

Seven abstracts of technical papers to be presented during the 1960 South Central Region Conference are given below. The conference will be held October 25-27 at the Mayo Hotel, Tulsa, Okla.

Co-chairmen of the six symposia in which the technical papers will be presented are listed below also.

Symposia Chairmen

Oil and Gas Pipeline: Chairman H. Stewart, Sun Pipe Line Co., Beaumont, Texas, Co-Chairman R. J. Emerson, El Paso Natural Gas Co., El Paso, Texas.

Oil and Gas Production: Chairman W. C. Koger, Cities Service Oil Co., Bartlesville, Okla., Co-Chairman J. A. Caldwell, Humble Oil & Refining Co., Houston, Texas.

Chemical Processing and Refining: Chairman Cecil Phillips, Jr., Humble Oil & Refining Co., Baytown, Texas, Co-Chairman E. N. Coulter, Cities Service Refining Corp., Lake Charles, La.

Protective Coatings and Plastics: Chairman H. D. Boggs, Fibercast Co., Sand Springs, Okla., Co-Chairman Eugene E. Donaldson, Pan American Petroleum Corp., Tulsa, Okla.

Water Handling: Chairman C. O. Smith Humble Oil & Refining Co., Tulsa, Okla., Co-Chairman Ray Amstutz, Earlougher Engineering, Tulsa, Okla.

Corrosion Survey and Testing: Chairman L. B. Nelson, Shell Pipe Line Corp., Houston, Texas, Co-Chairman L. G. Sharpe, Houston, Texas.

Technical Paper Abstracts

Glass Reinforced Plastic Pipe and Tubing in Chemical Service, by D. D. Kays, Frontier Chemical Co., Wichita, Kan.
Discusses physical and chemical deterioration encountered with glass reinforced pipe in chemi-

(Continued on Page 56)

1960 Corrosion Show Described in Color Slide Presentation

Color slides and a narrative script of the 1960 Corrosion Show held last March at Dallas during the 16th Annual NACE Conference are available for use as program material at section meetings.

The slide presentation, which can be given in 30 to 45 minutes, was designed to give those persons not attending the Dallas Conference an opportunity to see the diversity of corrosion control materials, services, processes and equipment displayed by the exhibitors.

Section program chairmen or other interested persons can arrange bookings on the 1960 Corrosion Show slides by contacting G. A. Rolak, NACE Central Office, 1061 M & M Bldg., Houston 2, Texas. Bookings should be made as far in advance of the meeting date as possible.

These slides can be shown on any standard projector designed to handle 35 mm color slides. Two copies of the script are sent with the slides so that the projectionist and narrator can make a smooth, well coordinated presentation.

1961 Corrosion Show Slides To Be Available

Program chairmen making plans for section meeting next year can schedule a showing of the color slides of the 1961 Corrosion Show to be held March 14-16 in conjunction with the 17th Annual NACE Conference at Buffalo, N. Y.

The 1961 Corrosion show slides will be available for section meetings and other interested groups after April 24, 1961. Bookings must be received at NACE Central Office by March 1.

Sections can schedule this slide presentation as part of a meeting at which a review of the Buffalo Conference is to be made.

Canadian Region

Edmonton Section had 28 members and guests at the June 23 meeting. Guest speaker was Matt Gould of Hagan Corporation Ltd. His topic was corrosion and scale problems in water systems.

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The 1960 Corrosion Control Short Course sponsored by the NACE Central Oklahoma Section will be held Sept. 28-30 at the University of Oklahoma, Norman.

Western Region

Nuclear and Missile Symposium to Be Included at San Francisco Conference

Abstracts on some of the 23 papers scheduled for presentation during the October 6-7 Western Region Conference are given below. Seven technical symposia have been scheduled for the conference which will be held at the Sheraton-Palace Hotel in San Francisco. Time schedule for the symposia also is given. Additional information on the conference was published on Page 68 of the June issue and Page 69 of the July issue of CORROSION.

A schedule of the Corrosion Control Short Course to be presented October 3-5 in conjunction with the Western Region Conference is published on Page 47 of this issue. The short course will be held at the University of California Extension Center, 55 Laguna St., San Francisco. It will be sponsored by the University of California at Berkeley and Los Angeles in cooperation with NACE.

General Corrosion Symposium
10:30 am-12:30 pm, October 6

Chairman: George E. Moller, Union Oil Company of California, Rodeo, Cal.

Alloy Selection, Heat Treatment and Economic Considerations Involved in Prevention of Stress Corrosion Cracking of Austenitic Stainless Steels, by Richard A. White, Bechtel Corp., San Francisco, Cal.

Divided into two parts: First part shows how alloy content was varied to prevent stress corrosion cracking. Data presented shows cracking times in 42 percent $MgCl_2$ with varying C, N, Ni and Si contents. Second part discusses engineering and economics of stress relief heat treatment. Cracking times of steel with different heat treatments are shown. Advisability of stress relief is discussed in light of data presented.

Stress Corrosion of Stainless Steel, a film produced by Mars G. Fontana and staff, Ohio State University Metallurgical Department.

Petroleum Processing Symposium
2-5 pm, October 6

Chairman: J. T. Patrick, Union Oil Company of California, Rodeo, Cal.

Sulfur in Flue Gas Corrosion, by Paul D. Miller, Earl L. White and Warren E. Berry, Battelle Memorial Institute.

Presents experimental results for corrosion by SO_2 in flue gas. Discusses relation of sulfur percentage, dew point and temperature on corrosion process.

Service Experiences of a Fired Heater in a Catalytic Desulfurizer, by Dannier Emanuelson and E. H. Ehmke, Tidewater Oil Co., Associated, Cal.

Deals with stress rupture failure of 9 percent chromium heater tubing which failed after excessive firing and coke build up. Metallurgical study provides temperature reached and time for rupture. Tubing was heat-treated in place as result of rapid shut down and fire fighting.

High Temperature Corrosion Problems in the Petroleum Industry, by G. R. Prescott and R. B. Setterland, C. F. Braun & Co., Alhambra, Cal.

Covers two areas of corrosion: First iron-aluminum and iron-aluminum-chromium alloys tested in hydrogen sulfide at various temperatures and concentrations. Corrosion characteristics are compared to stainless steels. Second, accelerating influence of welding slag on high temperature corrosion of alloys in flue gas containing varying amounts of sulfur.

Nuclear and Missile Symposium
2-5 pm, October 6

Chairman: W. L. Pearl, General Electric Company, San Jose, Cal.

Stress Corrosion of 410 Stainless Steel in Fuming Nitric Acids, by L. L. Gilbert, Aerojet-General Corp., Azusa, Cal.

Corrosion of Nickel Base Alloys in High Temperature Nitrogen Environments, by G. W. Titus and J. S. Brunhouse, Aerojet-General Nucleonics, San Ramon, Cal.

Corrosion in Water of Aluminum and Uranium—Implications in Power Reactor Design, by J. E. Draley, Argonne National Laboratory, Argonne, Ill.

Coatings, Linings and Plastics Symposium
Part 1: 2-5 pm, October 6

Chairman: Martin K. Weber, Shell Development Company, Emeryville, Cal.

Non-Solvent Coatings for Corrosion Control, by William Hodges, Hodges Chemical Co., Mountain View, Cal., and J. J. Bogner, Chemical Process Co., Redwood City, Cal.

Discusses two newly developed plastic coating materials and processes which do not involve solvent escape or entrainment, thereby reducing fire hazard, voids, inconsistencies and shrinkage normally associated with coatings containing solvents. Also discusses improved properties and chemical resistance, over-all performance as compared with solvent systems and new application equipment required.

Epoxy and Polyester Resin Applications in Production and Maintenance, by Claude B. Smith, Claude B. Smith Company, Oakland, Cal.

Study is given of the background necessary to determine which resin formulation should be considered and whether the application should be undertaken by plant personnel or by contract companies specializing in coating field. Outlines repair work to pipes, vessels and process equipment. Case history data will be presented.

Painting Practices on State-Owned Toll Bridges, by Dale Downing, California State Toll Bridge Authority, Oakland, Cal.

Underground Structures Symposium
9 am-12 noon, October 7

Chairman: Glynn Reineman, Pipeline Protection Company, San Francisco, Cal.

Continuous Recording of Pipe-to-Soil Potentials vs Distance, by Robert L. Davis, Superior Oil Co., South Pasadena, Cal.

Cathodic Protection and Zinc Grounding—Industrial Plant, by J. D. Ghesquiere and J. A. Michelson, Hinchman Corporation, Detroit, Mich.

Relates corrosion and grounding problems in construction of a large manufacturing facility. Discusses elimination of copper ground rods which cause galvanic corrosion and unusual cathodic protection current requirements encountered. Reviews design considerations for cathodic protection system installed during construction and selection of zinc rods for grounding. Test data obtained after completion of protection system are given also.

Design Construction and Operation of Underground Corrosion Control System — Glasgow AFB, by Robert T. Mercer, Consulting Engineer, Seattle, Wash.

Describes a survey of corrosive history of the site. Also gives design considerations for corrosion resistant materials, current requirements, and types of ground beds: deep weel, regular and sacrificial. Construction features are discussed, then operation of the system is reviewed.

Coatings, Linings and Plastics Symposium
Part 2: 9-12 noon, October 7

Maintenance Painting for Industrial Atmospheres, by Frank T. Radecke, Shell Oil Co., New York City.

Discusses variables that engineer must consider for an industrial painting project such as environment, material selection, degree of surface preparation, coating system, dry film thickness and methods of application. Also discusses subsequent repainting at proper intervals for economical painting maintenance program.

Prestressed Brickwork, by Robert R. Pierce and Allen M. Smith, II, Penn Center, Philadelphia, Pa.

Describes the old technique of bricklining of process vessels and towers to achieve a lining superior to standard brick or metalclad linings. Economic case history of first such lining in USA is given to illustrate performance of a high temperature chlorination tower.

A Survey of Recent Corrosion Resistant Resinous Coatings, by Paul E. Christianson, Walter N. Boysen Company, Oakland, Cal.

Water Treatment and Water Inhibition Symposium
2-5 pm, October 7

Chairman: B. A. Kronmiller, Nalco Company, Alameda, Cal.

Use of Inorganic Inhibitors for Corrosion Control in Water Treatment Systems, by E. F. Duffek, Stanford Research Institute, Menlo Park, Cal.

Discusses chromate and phosphate inhibitors to explain their function in treatment systems. Briefly reviews factors determining choice of inhibitor with emphasis on laboratory and field results.

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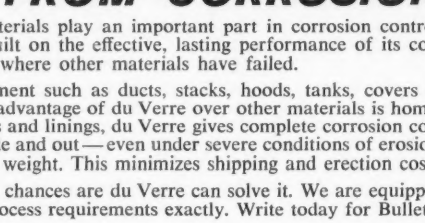

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Organic Material as Corrosion Inhibitors in Aqueous Media, by R. A. Powers, Union Carbide Consumer Product Co., Parma, Ohio. Describes function of organic materials as metal corrosion inhibitors, their mechanisms and factors determine the ability of these materials to form adsorbed or physical films on surfaces. General phenomena is described in which polar-type oils and amines promote a reaction between ferrous metals and dissolved oxygen which lead to electrochemical passivation of the metal surface.

Quantitative Evaluation of Corrosion in Water Supply Distribution Systems, by Werner Stumm, Harvard University, Cambridge, Mass. Quantitative evaluation of corrosion in water supply distribution systems should depend on long term investigations of corrosion and carrying capacity of pipes and water quality. Describes recently developed rapid electrochemical method using measurement of current required to polarize a corroding specimen a few millivolts.

Chemical Industries Symposium
2-5 pm, October 7

Chairman: R. L. Piehl, Standard Oil Company of California, Richmond, Cal.

Problems in Pilot Plant Corrosion Testing, by R. S. Treseder, Shell Development Co., Emeryville, Cal. Discusses problems involved in obtaining reliable corrosion data from pilot plant testing program. Subjects include material selection, specimen test locations and evaluation of experience and specimen data. Emphasizes two factors for consideration: variations in operating conditions between pilot plant and full scale plant and effect of differences in surface to volume ratio.

Effects of Molding Materials on Corrosion Resistance of Alloy Casting, by Dean D. Burgan, Electric Steel Foundy Co., Portland, Ore.

Stress Corrosion Cracking of Carbon Steel in Calcium-Ammonium Nitrate Solutions, by E. H. Edwards and R. L. Piehl, Standard Oil Company of California, and A. E. Barta, California Spray-Chemical Corp., Richmond, Cal. Discusses stress and corrosion cracking of carbon steel in calcium-ammonium nitrate fertilizer solution with respect to field experience and laboratory test results. Survey of failed storage tanks reveals cracking has been limited to heat affected zones of non-stress relieved welds. Shows that probability of cracking welded vessels is greatly reduced, if not eliminated, by stress relief after fabrication at temperatures from 950 to 1150 F.

San Joaquin Corrosion Tour To Be Held October 12-13

The study of corrosion problems and their control as experienced in petroleum producing and refining, utilities and agricultural industries will be made at the San Joaquin Valley Section Corrosion Tour to be held October 12-13. Headquarters will be at the Bakersfield Inn, Bakersfield, Cal.

Fee for the tour will be $15, which will include two lunches, a Luau dinner at the end of the tour and bus travel throughout the tour.

Pre-registration information can be obtained from Jack Penner, 305 E. Brundage Lane, Bakersfield, Cal.

Many projects visited two years ago will be re-visited with case history data available to show what control measures have been applied.

Some of the exhibits to be visited include gasoline plant vessels, gas gathering systems, storage tanks, etc. Other points of interest are inhibition treatment, cathodic protection, pipe line and tank interiors, coatings, water disposal systems, bacteriological treatment and others.

Chairman of the tour is Jerry Mitchell of Jones & Keller, Inc. Additional information on the tour can be obtained from the publicity chairman, Nat G. Haseltine, Standard Oil Company of California, Taft, Cal.

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Central Arizona Section heard Frank Buck of the Arizona Public Service Company speak on the causes and prevention of corrosion at the June 22 meeting.

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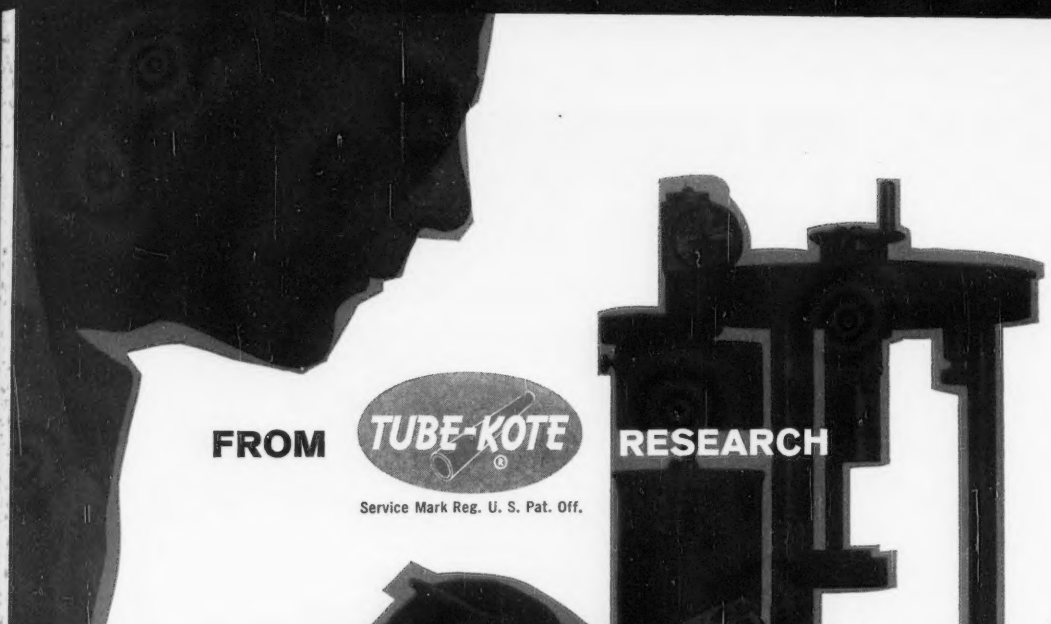

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NATIONAL and REGIONAL MEETINGS and SHORT COURSES

1960

Oct. 6-7—10th Annual Western Region Conference, Sheraton-Palace Hotel, San Francisco, Cal.

Oct. 6-8—Southeast Region Conference, Dinkler-Plaza Hotel, Atlanta, Ga.

Oct. 11-14—Northeast Region Conference. Prichard Hotel, Huntington, W. Va.

Oct. 19-20—North Central Region Conference. Pfister Hotel, Milwaukee.

Oct. 25-27—South Central Region Conference. Mayo Hotel, Tulsa.

November 14-16—Eastern Division, Canadian Region Conference, Hotel Royal, Montreal.

November 17-18—NACE Board of Directors Meeting, Sherman Hotel, Chicago, Ill.

1961

March 12—NACE Board of Director's Meeting, Hotel Statler, Buffalo, N. Y.

March 13-17—17th Annual Conference and 1961 Corrosion Show, Buffalo, N. Y., Hotel Statler.

March 17—NACE Board of Director's Meeting, Hotel Statler, Buffalo, N. Y.

Oct. 4-6—Western Region Conference, Hotel Multnomah, Portland, Oregon.

Oct. 9-11—North Central Region Conference, St. Louis, Chase Park Plaza Hotel.

Oct. 24-27—South Central Region Conference and Exhibition, Shamrock-Hilton Hotel, Houston.

Oct. 30-Nov. 2—Northeast Region Conference, New York City, Hotel Statler.

Southeast Region Conference, Miami, Fla., in conjunction with Miami Section's short course.

1962

March 19-23—18th Annual Conference and 1962 Corrosion Show. Kansas City, Municipal Auditorium.

October 1-4—Northeast Region Conference, Hotel Sheraton Ten Eyck, Albany, N. Y.

October 9-11—North Central Region Conference.

October 16-19—South Central Region Conference and Exhibition, Hilton Hotel, San Antonio.

SHORT COURSES

1960

September 12-16—Short Course on Process Industry Corrosion, sponsored by NACE Technical Committee T-5 and Ohio State University, to be held at Ohio State University, Columbus.

September 28-30—Central Oklahoma Section 1960 Corrosion Control Short Course, at University of Oklahoma, Norman.

October 3-5—Corrosion Control Short Course, sponsored by Western Region and University of California, San Francisco.

October 12-13—San Joaquin Valley Section Biannual Corrosion Tour Registration at Bakersfield Inn, Bakersfield, Calif.

November 7-11—Annual Florida General Conference Corrosion Short Course, Key Biscayne Hotel, Miami. Sponsored by Miami and Jacksonville Sections.

Free Radicals May Be New Source of Useful Energy

Free radicals may be a new source of useful power for man's increasing energy requirements.

Free radicals are molecular fragments which are known to possess very high energy potentials during their normally fleeting existence. In experiments conducted by the newly formed Energy Conversion Laboratories, 14121 West McNichols Road, Detroit 35, Mich., constant streams of the highly reactive particles are being produced by various means in a safe, simple and seemingly self-sustaining system. Their natural atomic reactions are being controlled at near room temperature. Their bursts of chemical energy are being converted directly into electricity and other forms of power.

Possible applications for free radical energy converters are equipment for desalting sea water, stationary and portable power generators and small packaged power plants for operating boats, trucks, tractors and other types of vehicles including automobiles.

Alternate Offices Built In Case of Nuclear Attack

In case of an atomic or nuclear attack on the New York City area, Standard Oil Company of New Jersey has built alternate headquarters outside New York in Morris County, N. J.

The headquarters provide remote storage of all vital corporate records, emergency accommodations for 130 persons, communications network including telephone, teletype and two-way radio circuits, standby electrical generator in case of general power failure and an artesian well for an independent water system.

A safety water spray with revolving spray heads was installed on the roof to wash down accumulations of fallout in case of an H-bomb attack.

Corrosion Magazine Binders Available From NACE Office

Keeping copies of CORROSION for permanent reference will be easier if sufficient interest is shown in the purchase of binders. The binders, being offered by CORROSION to its readers at a nominal cost, will hold 12 issues. They permit the copies to be kept on desks or in book cases without the hazard of being lost, misplaced or "borrowed."

The advertisement below gives a description of the binders and includes an order coupon. If sufficient orders are received before October 1 to indicate that the project is worthwhile, binders will be stocked and shipped from CORROSION's 1061 M & M Bldg., Houston 2, Texas office.

Alloy Casting Literature

A new booklet titled "Technical Publications List" which summarizes the results of years of research on high alloy castings has been issued by the Alloy Casting Institute, 1001 Franklin Ave., Garden City, N. Y. The booklet lists 46 technical papers, articles, reprints and data sheets resulting from the Institute's informational program.

Are You Keeping Corrosion on File?

Would you like a binder that will permit you to keep issues in sequence?

Provided there is sufficient interest by CORROSION readers, binders that will hold 12 issues will be available at nominal cost. Issues are held securely by a steel wire that fastens into the binder. This permits you to keep the binder upright on your desk, or in a book case. Copies are not damaged by the attachment and may be removed for permanent sewed binding if desired. This offer is contingent on a sufficient number of orders being received by CORROSION before October 1.

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City Zone State

South Central Region

(Continued From Page 51)

cal waste disposal well tubing service. Explains method of eliminating the exposure. Presents success and failure of this type pipe in various chlor-alkali plant services with comparisons to other materials. Defines areas of acceptable application for this pipe in handling chemical streams occurring in a chlor-alkali plant.

Corrosion Resistance of Refractory Metals at Elevated Temperatures, by John R. Schley, Haynes Stellite Co., Kokomo, Ind.
Presents brief resume of historical background and methods of manufacture and fabrication of tantalum, columbium, tungsten and molybdenum. Gives details on their corrosion resistant properties under wet conditions and at elevated temperatures.

Zinc and Lead-Silver Anodes Solve Several Refinery Corrosion Problems, by George E. Moller and John T. Patrick, Union Oil Company of California, Rodeo, Cal., and John W. Caldwell, Bunker Hill Company, San Francisco, Cal.
Describes four applications of cathodic protection in a petroleum refinery. These are new solutions to old problems. One is use of high purity zinc to protect water heels of four sour naphtha storage tanks. Second is use of lead-silver anodes to protect steel pipe coils in a salt water cooling box. Third and fourth involve use of zinc to protect a rotary salt water screen and a clarifier rake. Corrosive conditions are described, design basis given plus installation, results and economics of the systems.

Anodic Protection Against Sulfuric Acid Corrosion, by Carle E. Locke, Merle Hutchison and Norman L. Conger, Continental Oil Co., Ponca City, Okla.
Describes application of anodic passivation techniques to chemical process industry. Also describes fundamentals of passivation by application of anodic currents. Successful installations in oleum storage, sulfuric acid storage and sulfonation vessels are described. Data is given to show value of anodic protection in reducing corrosion rates, improving product quality by reduction of iron content and improvement in process efficiency due to elimination of troublesome emulsion.

Economy in Airless Spraying Vinyls, by Dwight L. DuPuy, Union Carbide Chemicals Co., Texas City, Texas.
Gives detail analysis of economics and advantages involved with principles of hydraulic spray painting.

Technology of Inorganic Zinc Coatings, by James B. Cox, Humble Oil & Refining Co., Houston, Texas.
Discusses physical properties of inorganic zinc coatings such as flexibility, abrasion resistance, heat resistance, adhesion and cathodic protection. Explains their resistance to aviation gasoline, jet fuels, aromatic hydrocarbons, ketones, esters and automotive gasolines.

Formulation and Application of a Bitumen Epoxy Coating for Pipelines, by Frank James, Jr., Allied Paint Mfg. Co., Tulsa, Okla.
Presents factors involved in formulation and application of a coal tar epoxy coating. Explains formulation of a hypothetical coating designed especially for pipelines.

•

Alamo Section had a showing of the International Nickel Company film "Corrosion in Action" at the June 21 meeting.

•

Permian Basin Section heard Jack L. Battle of Humble Oil & Refining Company speak on oil and gas well casing corrosion at the April 12 meeting. Total attendance was 72 members and guests.

Next scheduled meeting is set for September 13.

Guest speaker for the May 10 meeting was Alfred R. Erben of Sun Oil Company. His topic was laboratory and field studies of cathodic protection.

•

A Short Course on Process Industry Corrosion, sponsored by NACE Technical Committee T-5 and Ohio State University, will be held Sept. 12-16 at Columbus.

Region Plans Exhibition For 1961 Houston Meeting

An exhibition will be held concurrently with the South Central Region Conference at the Shamrock-Hilton Hotel in Houston, October 24-27, 1961. At the NACE board meeting in Dallas in March an exhibition was approved also to be held concurrently with the 1962 South Central Region Conference at the Hilton Hotel San Antonio, Texas, October 16-19.

•

The 1960 Corrosion Control Short Course sponsored by the NACE Central Oklahoma Section will be held Sept. 28-30 at the University of Oklahoma, Norman.

•

The 1960 South Central Region Conference will be held October 25-28 at the Mayo Hotel, Tulsa, Okla.

Annual Titanium Conference

The sixth annual titanium metallurgy conference sponsored by New York University's College of Engineering will be held September 12-13 at the University Heights campus in the Bronx. The conference is intended for engineers from industrial, research and governmental organizations.

The program will include discussions on the cryogenic behavior of titanium and its alloys, welding, heat treatment, alloying and fabrication techniques.

Additional information and applications can be obtained by writing Harold Margolin, New York University, University Heights 53, N. Y.

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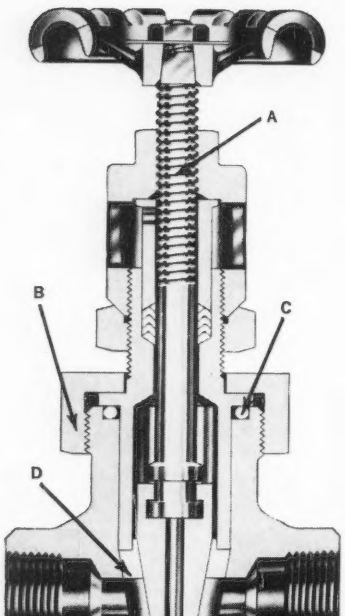

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North Central Region

Corrosion Problems of Four Industries To Be Discussed at Milwaukee Meeting

Twenty technical papers on corrosion control problems related to four industries will be given on the program for the October 19-20 North Central Region Conference, to be held at the Hotel Pfister in Milwaukee, Wis. Topics to be discussed are related to the process, utilities, brewing and pulp and paper industries of the North Central area of the United States.

Time schedule for presentation of these papers in the four technical symposia are given below. Abstracts are given for most of the papers.

Process Industries Symposium
Part One
9-12 noon, October 19

Chairman: Carroll Imhoff, Allis-Chalmers Mfg. Company, Milwaukee, Wis.

Corrosion Problems in Large Alkali-Chlorine Acid Plant, by Joseph Bosich, Diamond Alkali Co., Painesville, Ohio.
Discusses a few of many corrosion problems that occur in large multiple chemical plant. Describes practical corrective measures used in control of unusual corrosion problems. Briefly describes case histories of several problems.

Corrosion Resistance of Stainless Steel Type 329 in Nitric Acid Containing Chloride, by Irvine I. Tingley, Department of Mines and Technical Surveys, Ottawa, Canada.
Type 329 was found to have greater corrosion resistance than Types 316, 304L and 309SCB in condensing vapors from solutions 7.5N in nitric acid and to 0.38 N in chloride ion. Welded specimens of Type 329 must be fully annealed to prevent severe intergranular attack in heat-affected zone. Discusses mechanisms of this attack and influence of accumulated chromic and ferric ions. Also discusses investigation of severely stressed specimens of this steel in these solutions.

Some Corrosion Studies Involving Chemical Equipment, by H. W. Schmidt and A. A. Brower, Dow Chemical Co., Midland, Mich.
Presents unusual case histories of chemical processing environments requiring unique approaches to solve corrosion problems. One is an application of cathodic protection of nickel equipment in a high purity salt crystallization process using a specially designed magnesium anode system to eliminate product contamination. Another case describes laboratory study on polarization characteristics of steel in pyroligneous acid revealed that impressed current system could be used to protect steel storage tanks, current density being quite nominal. Another case describes rather simple material and design changes which successfully eliminated all galvanic effects in a salt brine filter unit. Also describes several problems involving hydrogen sulfide and/or sulfide scale which induced accelerated corrosion of pipelines and heat exchangers in raw brine service.

Massive De-Aluminization of Aluminum Bronze by Chloride Crevice Attack, by L. W. Gleekman, Wyandotte Chemicals Corp., Wyandotte, Mich., and R. K. Swandby, International Nickel Co., New York, N. Y.
Discusses failure of a wrought 3-foot diameter aluminum bronze drum used to dry a calcium carbonate paste. Failure was determined to be caused by de-aluminization. Ultimate solution was use of 90-10 Cupro-Nickel wrought plate welded with 70-30 Cupro-Nickel.

Utilities Symposium
Part One
9-12 noon, October 19

Chairman: Frank Stetler, Wisconsin Telephone Co., Milwaukee, Wis.

Gas Distribution System—Corrosion and Cathodic Protection Problems, by John O. Mandley, Michigan Consolidated Gas Co., Detroit, Mich.
Discusses corrosion organization of a gas distribution company and problems encountered in cathodic protection of large pipe distribution systems. Gives some hazards involved in distribution work.

Corrosion Aspects of Interconnecting the Telephone Aerial Cable Sheaths and the Power Multi-Grounded Neutral System, by Oliver Henderson, Ohio Bell Telephone Co., Cleveland, Ohio.
Discusses this interconnection as improving the rapid de-energization of power wires after a fault, reduces damage caused by lightning and increases personnel safety on pole lines. Discusses secondary aspects that are deleterious and which cannot safely be neglected or ignored. Describes causes of difficulties and suggests corrective action. Concludes that all aspects of problem can be reconciled.

Corrosion of Steam Turbines and Condensers in Central Station Power Plants, by A. Bayard Sisson, Commonwealth Edison Co., Chicago, Ill.
Discusses variety of corrosion problems on steam turbines and condensers involving numerous metals and alloys. Shows that these problems have been eliminated by appropriate practices although some must be tolerated and kept to a minimum by routine maintenance. Also discusses corrosion mechanisms involved. Summarizes operating experiences, reasons for corrosion and condition changes made to improve performance.

Observations of Design and Performance of Well Type Ground Beds, by Ted L. Canfield, Cathodic Protection Service, Tulsa, Okla.
Case histories are given of well type ground beds on different types of structures. Data indicates acceptable ground bed resistance, high degree of stability and good current distribution characteristics. Discusses design and economics of these ground beds and compares them to conventional surface type installations.

Brewing Industries Symposium
1:30-4:30 pm, October 19

Chairman: Don Westerman, Miller Brewing Company, Milwaukee, Wis.

Paints and Coatings in the Brewing Industry, by Kenneth Tator, K. Tator Associates, Coraopolis, Pa.
Outlines coating problem in brewery processes. Presents principles and classification of paint coating selection. Gives application and maintenance suggestions to insure maximum protective life of applied coatings. Also presents economics of protection.

Cathodic Protection of Glass Lined Storage Tanks, by D. D. Walczyk, A. O. Smith Corp., Milwaukee, Wis.
Discusses laboratory tests that proved the iron pick-up by beer during storage in a glass lined tank could be reduced to negligible amount with cathodic protection even if bare metal area in tank became excessive. Also was shown that simple and cheap graphite anode could be used without harming appearance or taste of the beer.

Process Industries Symposium
Part Two
9-11:45 am, October 20

Special Purpose Panels for Evaluation of Internal Barrier Coatings, by O. W. Siebert, Monsanto Chemical Co., Monsanto, Ill.
Describes techniques and panel designs for testing and evaluating chemical resistant linings and barrier coatings used for inside tanks. Emphasis is given to successful use of panels of several geometric shapes: Spheres, tori and mobius strips. Each of these designs is described with benefits derived from their specific use. Gives results of plant tests. Concludes that panels in configuration of a mobius strip provide a test system offering the most reproducible and useful data.

Composition of Corrosion Products Formed in Steam Condensate, by R. D. Eberhardt, E. W. Arneson and C. E. Imhoff, Allis-Chalmers Mfg. Co., Milwaukee, Wis.
Discusses compositions of corrosion products formed in laboratory on various commercial alloys in contact with condensed steam as identified by X-ray diffraction. Condensate contained controlled amounts of dissolved gases known to be present in power plant condensers, turbines and return lines. Experiments were conducted at 100 F with a pH of 6 and at 212 with pH values from 5.1 to 9.6.

(Continued on Next Page)

New Methods of Simulating Corrosive Plant Conditions in the Laboratory, by A. Orman Fisher, Monsanto Chemical Co., St. Louis, Mo. Describes recent laboratory techniques developed for reproduction of corrosive plant conditions. Typical studies involving heating and cooling surfaces under elevated temperature and pressure conditions are discussed. Corrosion data on various alloys in sulfuric acid, nitric acid, tall oil and other environments obtained in laboratory equipment are included for illustrative purposes.

Utilities Symposium
Part Two
9-11:45 am, October 20

Stray Current vs Non-Stray Current Corrosion Mitigation, by Vernon B. Pike, Bell Telephone Laboratories, Murray Hill, N. J.

Discusses corrosion attack on underground lead covered cables produced by stray current, how the trouble was resolved and how it was controlled. Also discusses transition as stray current ceases to be a trouble factor in maintenance of these cables. Also discusses factors influencing behavior of copper-copper sulfate reference electrode in this work and how to avoid errors from several causes.

Experiences in Evaluating Protective Materials for Utility Hardware, by John S. Melvin, McGraw Edison Co., Milwaukee, Wis.

Discusses choice of protective coating to fulfill qualifications. These involve such factors as cost, resistance to pertinent corrosion media, mechanical properties, ease of application, universal applicability to an entire product line, availability and customer acceptance. Discusses zinc hot dip galvanized coatings as a protective material most often satisfying these qualifications.

Devices Used in Corrosion Control of Power Cables, by J. J. Pokorny, Cleveland Electric Illuminating Co., Cleveland, Ohio.

Describes various devices used to control power cable corrosion, with emphasis on improvements such as the electrolysis switch, test current interrupter and cathodic protection and forced drainage rectifiers. Use of silicon power rectifiers as an electrolysis switch is described. Also gives method of grounding and corrosion protection of a pipe cable system.

Pulp and Paper Symposium
3-5 pm, October 20

Chairman: Steve Baisch, S. J. Baisch Associates, Kaukauna, Wis.

Corrosion Problems from the Insurance Viewpoint, by H. M. Canavan, Mutual Boiler and Machinery Insurance Co., Waltham, Mass.

Briefly summarizes general principles of corrosion including those in order of their relative economic preference in pulp and paper industry. Also includes brief introductory review of insurance coverages for boilers and pressure vessels used. Attention is given to relationship between insurance and corrosion. Slides used to illustrate property damage resulting from excessive corrosion.

Polarization Curve Technique for Studying Corrosion and Electro-Chemical Protection, by W. A. Mueller, Pulp and Paper Research Institute of Canada, Montreal, Canada.

Discusses experimental methods used in study of polarization curves. Relationship between gradient of curves and corrosion rate in uninhibited corrosion processes is investigated. Curve analysis is explained, and two methods of using curves in corrosion studies are compared. Conditions for stability of active and passive states are specified for application in anodic and cathodic protection.

Cathodic Protection of Pulp Digesters: A Proposed Solution for Vapor Phase Protection, by L. C. Watson, A. O. Smith Corp., Milwaukee, Wis.

Analyzes factors which are necessary for successful cathodic protection of vapor phase of a pulp digester and points out how each of various factors are being or have been met successfully in different systems. Shows how these various known systems can be adapted to protection of vapor phase of the digester and the liquid phase as well with a minimum of uncertainty in the results. Also points out methods by which the system can be applied with relatively low costs, both for first installation and for installations which have to be made when the anode structure has been made up.

•

Twin Cities Section heard M. Nicholson, Head of the Department of Metallurgy at the University of Minnesota, speak on the fundamentals of corrosion at the July meeting.

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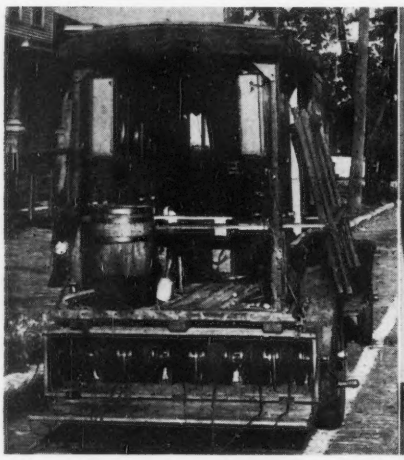

JOINT MEETING of the Ohio Valley Section and the Louisville Electrolysis committee was held May 20 to celebrate the 36th birthday of the Louisville Electrolysis Committee. The committee has been affiliated with NACE's T-7C Southeast Region Corrosion Coordinating Committee since 1957. Photograph at left shows one of the trucks used in 1926 for making some of the first electrolysis surveys in Louisville.

Foreign Corrosion Reports

Foreign corrosion study reports for 1959 from Japan, Switzerland and Germany have been prepared by the Inter-Society Corrosion Committee's Subcommittee on Relations with Foreign Organizations. Hugh P. Godard, immediate past president of NACE, is chairman of this ISCC subcommittee.

Corrosion Activity in Japan

Corrosion Committee of the Japan Society for Promotion of Science; K. Ishimura, Chairman.

Committee members, supporting members and associate members are numbering 60, 62 and 500 respectively. The committee meetings were held 11 times during the year of 1959 at which 27 reports on corrosion were presented. Publication of this group include "Corrosion Engineering" (12 issues per year). A four day lecture meeting was held in December at Tokuyama City, Shikoku.

A two-day corrosion symposium was held in November at which papers presented and subjects brought out for discussion were as follows:

1. Relationships between Faradaic impedance and polarization potential of metals.
2. Influence of H_2S, Fe^{++} and Fe^{+++} on the corrosion rate of steel during acid cleaning of reformers.
3. Study on amine type corrosion inhibitors, inhibitors with branched hydrocarbon radicals.
4. Corrosion resistance of zirconium in sulfuric acid at high temperature.
5. Corrosion phenomena of aluminum bronze.
6. Stress corrosion of cooling pipes in the oil containing transformer.
7. Corrosion of structural materials used for oil vessels of the tanker.
8. Study on corrosion of packing materials.
9. A simple and practical polarization meter.
10. Effect of polishing of the propeller surface on electro-potential of hulls and shafts of the ship.
11. Protective potential of copper alloy under a sea water cavitation.
12. Corrosion of lead alloy anode for cathodic protection.

Subjects discussed are listed below:

1. Standard protective potential and its measurement.
2. Relationships between cathodic protection and coating.
3. Pittings of stainless steel.
4. Stress Corrosion of stainless steel.
5. Practical uses of titanium as a corrosion resistant material.

Electric Corrosion Control Committee; S. Saheki, Chairman

The following subjects were taken up by the subcommittee for extensive studies:

1. Corrosion of electric railway.
2. Stray current corrosion.
3. Corrosion of lead cable sheaths.
4. Corrosion of underground iron and steel pipes.
5. Standardization of methods of cathodic protection and coating of underground pipe.

Japan Corrosion Engineering Association; S. Inouye, Chairman

The committee and a few subcommittees held meetings to standardize the measurement of corrosion of metals and the quality of corrosion preventive oils, vaporizable inhibitors and anti-corrosion packings. Meetings for lecture and a short course were held to popularize the knowledge on corrosion control of iron and steel products.

Electro-Chemical Corrosion Preventive Committee; G. Okamoto, Chairman

Committee meetings were held three times during 1959 to discuss theoretical aspects of electro-chemical corrosion.

Association of Surface Treatment of Metals; H. Hashimoto, Chairman

Meetings for lecture and a short course were held to promote practical knowledge on plating, metalicon and other surface treatments.

Light Alloy Association

Three subcommittees held meetings to study the surface treatment and cathodic protection of light alloys.

Shipbuilders Research Association

The committee held meetings every other month to discuss the corrosion protection of ships.

Inter-Society Corrosion Symposium

A three-day joint corrosion symposium was held in November, 1959, at Hyogo to discuss various corrosion problems under auspices of Japan Institute of Metals, Japan Chemical Society and Electro-Chemical Society at which 42 papers were presented for discussion.

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Corrosion Activities in Germany

The Sewage Association (Abwassertechnische Vereinigung) issued instructions on rust removal and painting of structural sections in sewers and sewage treatment plants.

Exhibitions with lectures were arranged in Denmark and Czechoslovakia by the Information Center for Seaworthy Packaging (Beratungsstelle fur seema Bige Verpackung) in which questions of corrosion were considered. The climatic condition existing in a ship's holds was investigated on a trip to East Asia.

The German Bunsen Society for Physical Chemistry (Deutsche Bunsen-Gesellschaft fur Physikalische Chemie) included in their 58th meeting six papers on the electrochemical basis of corrosion.

The German Society for Chemical Apparatus (Deutsche Gesellschaft fur Chemisches Apparatewesen or "Dechema") held, in their professional committee on surface protection, meetings of the seven subcommittees in which results of investigations in the following fields were discussed: Production and properties of coatings on metals; phosphates, chromates, synthetic materials, lacquers and enamels; brick linings and cement, and inhibitors.

The large compilation "Dechema-Werkstoffetabelle" (3rd edition) was

(Continued on Page 62)

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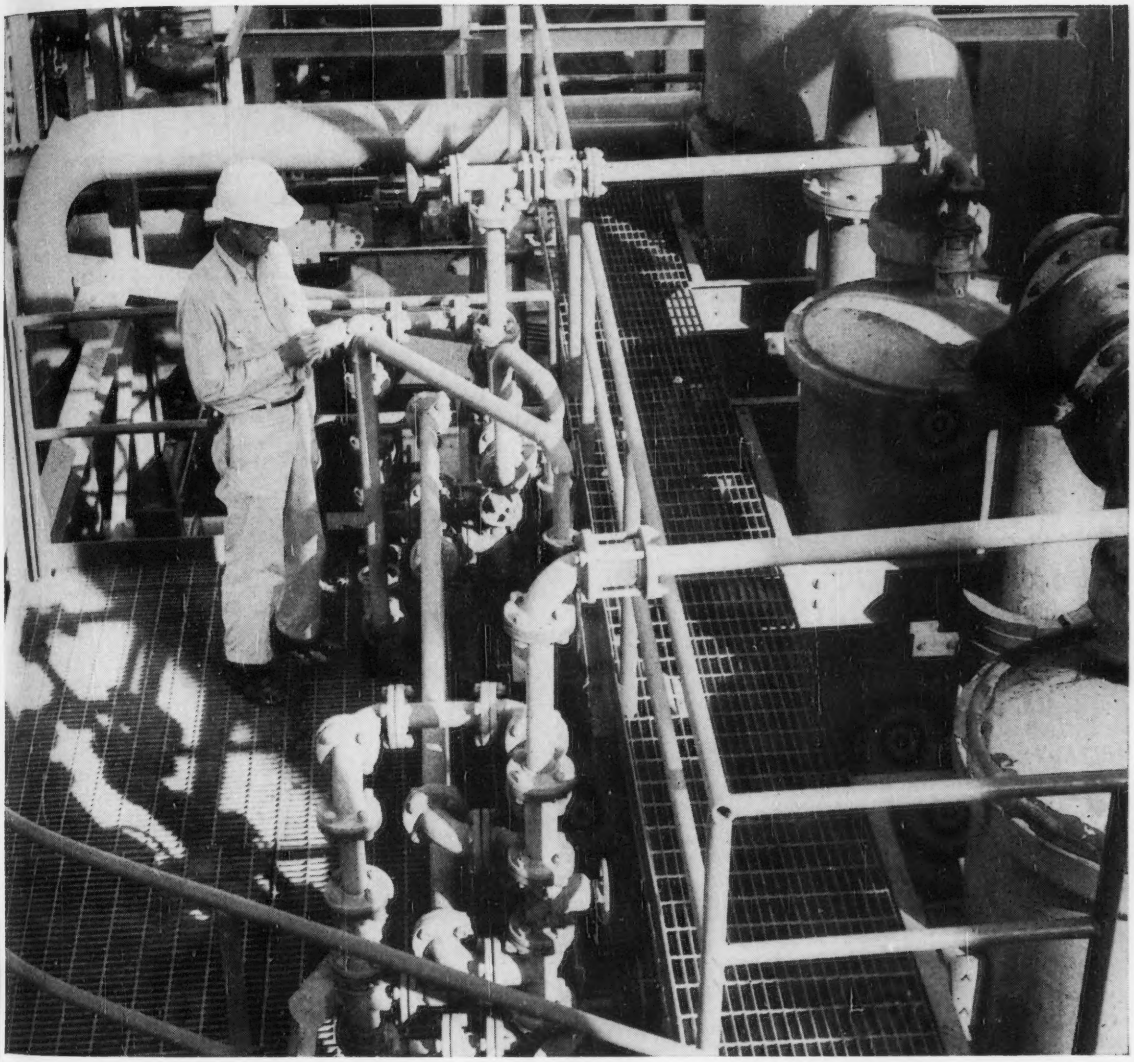

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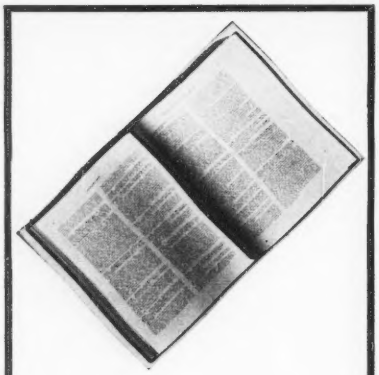

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Foreign Corrosion—

(Continued From Page 60)

continued to the extent of 900 pages which treat corrosion from abietic acid to magnesium oxychloride.

The German Society for Metallurgy (Deutsche Gesellschaft fur Metallkunde) held, with the German Iron and Steel Association (Verein Deutscher Eisenhuttenleute) a symposium on pitting corrosion with 14 papers. A book appeared on the 1958 symposium ("Cathodic Protection").

The German Society of Gas and Water Specialists (Deutsche Verein von Gasund Wasserfachmannern) held internal meetings on the effect of town gas drying on corrosion, the determination of aggressive constituents in soils, and the aggressivity of cold and hot water. In addition, the society was concerned with cathodic protection in soil, partly in collaboration with the German Electro-technical Society (Verband Deutscher Elektrotechniker).

The German Specification Committee, Subcommittee for Material Testing (Deutsche Normenausschuss, Fachnormenausschuss Materialprufung) published specifications or draft specifications on the testing of the resistance of enamel to hydrochloric acid, the determination of the pH value of paper, commercial phosphating methods, nickel salts for nickel baths, the determination of the thickness of zinc and tin coatings and standard climates for corrosion testing in artificial climates.

The Research Organization for Internal Combustion Engines (Forschungsvereinigung fur Verbrennugnskraftmaschinen) prepared a literature review on corrosion and cavitation in internal combustion motors by cooling water.

The German Chemical Association (Gesesellschaft Deutscher Chemiker) arranged through its corrosion committee for 22 corrosion research projects to be paid for by an annual contribution from the Federal Ministry of Economics, amounting to 430,000 DM. Their special group "Paints and Pigments" held a conference which included 13 papers and three discussion evenings. The "Water Chemistry" group was concerned with the resistance of asbestos cement to aggressive waters.

The Harbor Construction Society (Hafenbautechnische Gesellschaft) was concerned with the testing of the corrosion behaviour of water at the sea coast and in river mouths. They installed corrosion samples in harbors of various water compositions.

The Society of Shipbuilders (Schiffbautechnische Gesellschaft) considered methods of scale removal, anti-fouling paints and electrical behavior of zinc anodes on travelling ships. They published directives for flame spraying.

The Bituminous Coal Mining Society (Steinkohlenbergbauverein) was particularly concerned with the increased use of materials with high corrosion resistance in mining and with the possibility of determining accurately the portion of pure zinc in zinc coatings on hopper cars.

The German Iron and Steel Association (Verein Deutscher Eisenhuttenleute) worked on drafts for new specifications in which instructions were given for stainless steel in various applications such as milk pipes, heat exchanger tubes and tubes protecting thermometers. The Society continued previous atmospheric exposure tests and constructed a new weathering stand on Helgoland Peninsula (North Sea).

The Society of German Engineers (Verein Deutscher Ingenieure) published specifications on the corrosion protection of hot water heating installations and prepared corresponding specifications for hot water tanks.

G. Schikorr
Chemische Landesuntersuchungsantalt
Stuttgart N, West Germany
Klenestrasse 18

Swiss Publications on Corrosion Listed

Listed below are Swiss publications on metallic corrosion and corrosion protection from 1959.

W. Feitknecht. Studies on Influence of Chemical Factors on Corrosion of Metals. *Chemistry and Industry*, 1102 (1959).

W. Feitknecht. On Oxidation of Solid Iron Hydroxides in Aqueous Solutions. *Z Elektrochem*, **63**, 34 (1959).

W. Feitknecht and H. W. Lehmann. On Oxidation of Magnetite (Fe_3O_4) to γ —Fe_2O_3. *Helv Chim Acta*, **42**, 2035 (1959).

H. Kunzler and K. Vogtli. Problems of Led Cable Corrosion 9—The Corrosion Inhibiting Effect of Tars and Other Substances. *Tech Mitt PTT (Bern)*, **3**, 81 (1959).

K. Vogtli. Corrosion Tests With Different Types of Cable Under Natural Conditions. *Tech Mitt PTT (Bern)*, **10**, 430 (1959).

L. Piatti, A. Kung and A. Kaiser. Surface Wetting of Engineering Material. *Werkstoffe u. Korrosion*, **10**, 239 (1959).

A. Bukowiecki. Studies on Corrosion Behavior of Non-Drying Oils and Fats With Special Regard to Modern Lubricants and Protective Materials for Metals. *Schweiz Arch angew Tech*, **25**, 23, 62, 96, 126 (1959).

A. Bukowiecki. Same study as above published as Report No. 191 of the Swiss Federal Laboratory for Testing Materials and Research.

R. Larson. On the Beneficial Effects of Zinc Di-Thiophosphates in Modern Motor Oils on Corrosion and Wear. *Schweiz Archiv angew Wiss Tech*, **25**, 54 (1959).

A. Bukowiecki. Investigations on Corrosion Behavior of Aluminum and Aluminum Alloys in Organic and Inorganic Bases. *Werkstoffe u. Korrosion*, **10**, 91 (1959).

A. Bukowiecki. On Corrosion Behavior of Metals in Non-Aqueous Liquids. *Metall (Berlin)*, **13**, 536 (1959).

A. Bukowiecki. Research and Experience on the Effects of Washing Agents on Metals. *Seifen-Ocle-Fette-Wachse (Augsburg)*, **85**, 621,653 (1959).

M. Hochweber. Comparison of Rapid Test Methods for Paint Coatings With Service Behavior. *Deutsche Farben-Zeitschrift*, **13**, 218 (1959).

H. Kunzler, Grounding and Potential Connections in Tank Systems With Respect to Corrosion Protection. *Monatsbull Schweiz Ver Gas u Wasserfachmn*, **39**, Nr. 10, (1959.

E. Brandenberger
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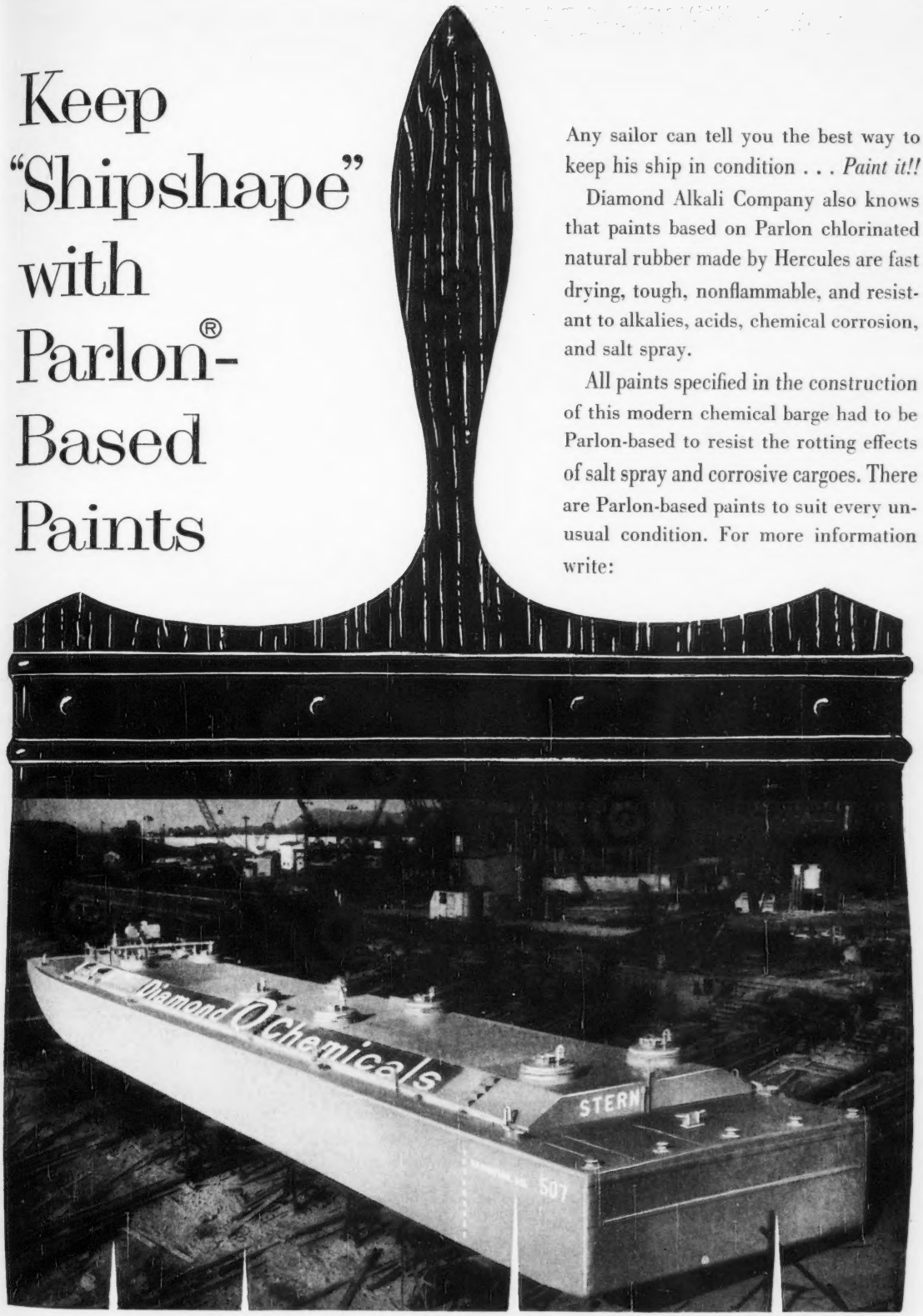

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CORROSION ABSTRACTS

INDEX TO CORROSION ABSTRACTS

Vol. 16 August, 1960 No. 8

2. TESTING
- 2.1 General 65
- 2.2 On Location Tests 65
- 2.3 Laboratory Methods and Tests 65

3. CHARACTERISTIC CORROSION PHENOMENA
- 3.2 Forms 70
- 3.3 Biological Effects 71
- 3.4 Chemical Effects 71
- 3.5 Physical and Mechanical Effects .. 71
- 3.6 Electrochemical Effects 72
- 3.7 Metallurgical Effects 73
- 3.8 Miscellaneous Principles 74

2. TESTING

2.1 General

2.1.1, 3.2.2, 6.4.2, 4.6.11

Pit Depth Measurements as a Means of Evaluating the Corrosion Resistance of Aluminum in Sea Water. T. J. Summerson, M. J. Pryor, D. S. Keir and R. J. Hogan. American Society for Testing Materials, Special Technical Pubn. No. 196, 157-175 (1957).

The development of a quantitative method for evaluating the resistance of aluminum to pitting corrosion in sea water is described. The method is based on a statistical treatment of penetrometer measurements of every pit present on test specimens immersed in sea water for periods up to 24 months. Normal frequency distributions were obtained by plotting the frequency of pitting against the square root of the pit depth. Calculations of the mean square root pit depth, the standard deviation, and standard error of the mean square root pit depth are shown. The importance of the mean square root pit depth, the standard deviation, and the frequency of pitting in determining the manner by which pitting corrosion proceeds is discussed. A sampling procedure method is described wherein quantitative data on the resistance of aluminum to pitting corrosion in sea water can be obtained within useful limits of confidence by statistical analysis of measurements of only a fraction of the total number of pits present. (auth)—ALL. 16293

2.1.2, 2.3.7

Coating Evaluation and Laboratory Correlation with Field Experience. W. N. B. Armstrong and J. W. Suggitt. Chem. in Canada, **9**, No. 11, 45-47 (1957).

The purposes of laboratory coating tests and methods used to control factors affecting reproducibility are presented. The logical interpretation of results is discussed. Examples are then given where laboratory evaluations have been used to provide suitable coatings and technique for field use. The misinterpretation of test data allowing the use of finishes with unsatisfactory performance characteristics is demonstrated. Finally, it is shown how laboratory data have been extended to limited field tests and eventually to full scale coating applications.—RPI. 15524

2.2 On Location Tests

2.2.2, 5.4.5, 4.2.5, 8.9.5

Zinc Oxide in Ships' Paints: Three-Year Marine Exposure Tests. A. Pass and E. A. Fishwick. J. Oil Col. Chem. Assoc., **41**, No. 9, 653-675 (1958).

The paper gives the results of a series of co-operative paint exposure tests of ships' topside paints exposed under marine conditions at sites in Great Britain, Sweden and North America. The primary object was to compare the behavior of alkyd paints containing zinc oxide as a major pigment with similar zinc-free paints. Opportunity was taken to use several types of zinc oxide as varying fractions of total pigment, to alter the extender and to compare pigmentations of the types used by the U. S. and British Navies. The paper discusses in detail the relative site severities and the particular types of paint failure encountered during an exposure of three years. Differences between the paints tested were negligible after 6 months, and small even after two years' exposure. During the third year's exposure the differences in the chalking properties of the various paints became more pronounced, particularly in the case of the zinc-free paints. The tests demonstrated that alkyd paints of the type tested and containing zinc oxide can be used satisfactorily under marine conditions with a life of up to three years. They compare favorably with similar paints made without zinc oxide and are much cheaper. 3 tables, 6 figures.—RPI. 17525

2.2.2, 6.2.3, 6.2.4

Corrosion of Metals in Tropical Environments. Part 2. Atmospheric Corrosion of Ten Structural Steels. C. R. Southwell, B. W. Forgeson and A. L. Alexander. U. S. Navy Dept., Naval Research Lab. Report No. 5002, December 11, 1957, 16 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington, D. C. (Order PB 131175).

Comprehensive corrosion data for 10 structural steels exposed to inland and marine tropical atmospheric environments were evaluated following an 8-year exposure period. Corrosion rates of unalloyed low-carbon and low-alloy steels exposed in the marine atmosphere are compared with those of similar steels following exposure to marine atmosphere at Kure Beach, North Carolina. Comparison of corrosion damage for a mild carbon steel exposed with millscale and with a pickled surface is shown. Tropical corrosion rates of unalloyed carbon steel, low-alloy steels containing chromium and nickel, and 4 proprietary low-alloy steels are compared with that of copper-bearing steel. 2 and 5 nickel steels were very effective in resisting corrosion. After 8-year exposure nickel steels were corroded 45-59 percent less than copper-bearing steel. Corrosion resistance of chromium steel was comparable to that of nickel steel. Tables, photos, graphs.—INCO. 16287

2.2.2, 6.4.2

Results of Atmospheric Corrosion Tests on Aluminum and Its Alloys in Italy. (In French.) Eugenio Hugony and Giuseppe Luft. Rev. Aluminium, **34**, 379-393 (1957) April.

Factors considered include changes in the external aspect, reduction of the mechanical properties, and alterations in the microstructure.—BTR. 14682

2.3 Laboratory Methods and Tests

2.3.2, 5.3.4

The Corrosion Resistance of, and an Evaluating Method for, Various Electrodeposits on Steel. H. W. Dettner. Plating, **46**, No. 5, 469-480 (1959) May.

The article describes tests of the corrosion resistance of electroplated zinc, cadmium, tin and tin-zinc alloys of 2, 5 and 10μ thickness. Salt spray, humidity, sulfur dioxide/carbon dioxide, and outdoor exposure tests were used. The percentage of rust area after the conclusion of the test was used to obtain numerical data, but the exposure time to the first indication of corrosion products was also noted. Comparisons of the

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value of the various coatings under different conditions are made and in general it was found that the zinc and cadmium coatings had similar corrosion resistance, and both were superior to tin. The tin-zinc alloys were intermediate, except in the salt spray test where the alloy rusted very slowly. 4 figures. 9 tables.—ZDA. 19085

2.3.2, 2.3.4, 5.3.2

The Testing and Examination of Electrodeposits. Pt. III. Salt Spray Testing. R. Quarendon. Product Finishing, **10**, 62-71 (1957) Aug.

Immersion tests; assessing results; applications and short-comings; operational techniques; A.R.E. and intermittent spray tests; spray solutions; "Corrodkote" and sulfur dioxide tests.—BTR. 14796

2.3.2, 5.3.2

Salt Spray Testing of Tinplated Copper. Pt. II. Comparison of Corrosion Rates in Different Test Locations. Martin S. Frant. Plating, **45**, 734-739 (1958) July.

By chemical porosity tests from 15 different test locations differences in corrosion results were found between different cabinets, and within individual cabinets, thus indicating the need for additional work on test variability.—BTR. 16476

2.3.2

Corrosion Testing as an Engineering Auxiliary. D. J. Fishlock. Mech. World, **138**, No. 3469, 342-348 (1958) August.

Describes very simply methods of testing the resistance to corrosion of paints and metal coatings on steel. It discusses the preparation of test samples, atmospheric exposure, various salt-spray tests, the use of humidity cabinets, accelerated weathering cycles and other quick methods of evaluating coatings. In each case, brief details are given of apparatus, operating conditions, procedure and limitations. 10 figures.—ZDA. 16522

2.3.6, 6.3.5, 3.2.3

The Microtopography of Oxide Films on Niobium. J. V. Cathcart, J. J. Campbell and G. P. Smith. Oak Ridge National Lab. J. Electrochem. Soc., **105**, 442-446 (1958) August.

An electron microscopic study of the oxide films formed on niobium permits the apparently anomalous oxidation rate behavior of niobium to be explained in terms of the formation of small, blister-like cracks in the oxide film. An oxidation model, based on the idea that an oxidation process which is maintained by interstitial-anion or anion-vacancy diffusion leads to the generation of stresses in the oxide film, is proposed to account for these results. The observation of similar blister-like cracks in oxide films on titanium is also reported. (auth)—NSA. 16798

2.3.5, 3.2.3

A New Experimental Technique for the Study of Films Produced at Electrochemical Interfaces. B. D. Cahan and P. Rüetschi. The Electric Storage Battery Co. J. Electrochem. Soc., **106**, No. 7, 543-547 (1959) July.

A new experimental technique for the study of the corrosion films at electrochemical interfaces has been developed. It consists of applying a square wave current superimposed on a constant d-c current. The a-c current charges and discharges the double layer capacity at a high frequency. From the voltage transients produced by this current, as observed with an oscilloscope, the nature of the electrode interface can be studied. The instrumentation is described in detail, and experimental results for corrosion films on lead are presented. Capacities were measured with a precision of better than 3 percent and voltages to 1 mv during transients lasting only a few seconds. 18301

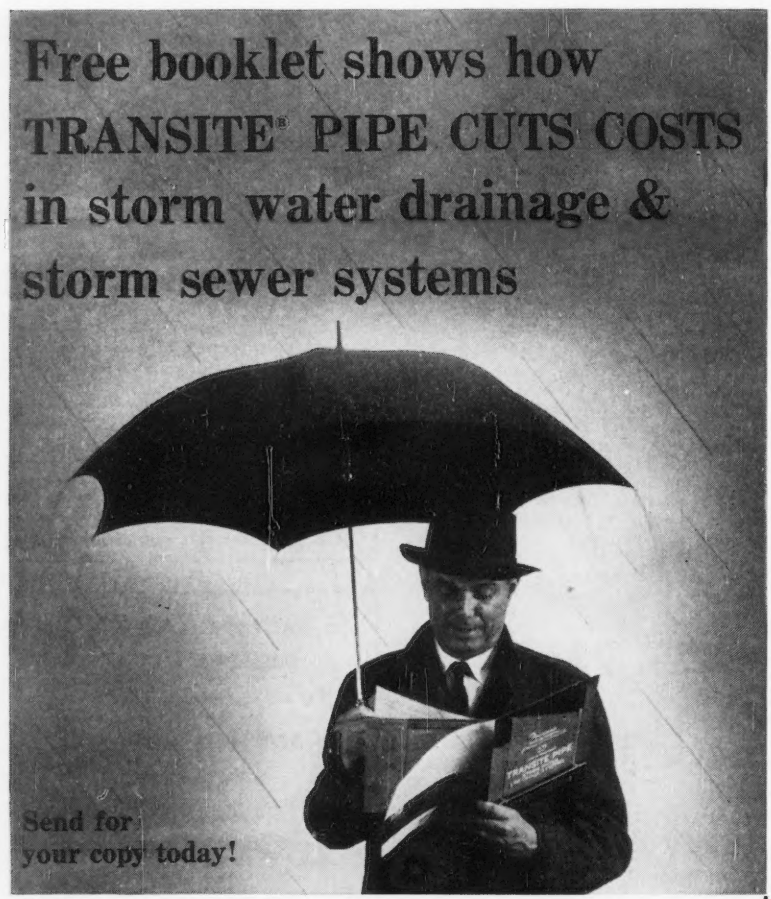
2.3.5, 5.4.5, 3.8.4, 4.3.6, 4.6.11

Electrical Measurements of Immersed Paint Coatings on Metal. Pt. II. Effect of Osmotic Pressure and Ionic Concentration of Solution on Paint Breakdown. D. M. Brasher and T. J. Nurse. J. Applied Chem., **9**, Pt. 2, 96-106 (1959).

Electrical capacitance and electrode potential measurements have been used to study the behavior of painted steel panels immersed in solutions of sucrose and of sodium chloride and other electrolytes, and in sea water. The water uptake of the paint in the early stages of immersion depends only on the osmotic pressure of the solution. The time to reach breakdown of the paint in sodium chloride solutions depends primarily on water uptake and secondarily on ionic concentration. The time to reach breakdown in sea water is considerably greater than that in sodium chloride solutions of similar ionic concentration. Corrosion rate after breakdown is primarily a function of the anodic/cathodic pore resistance, which is related to the electrolytic conductivity of the ambient solution; it is decreased in concentrated solutions by the diminishing solubility of oxygen. A mechanism of the course of paint breakdown is suggested.—RPI. 18317

2.3.9, 6.3.20

Thickness Measurements of ZrO_2 Film on Corrosion Tested Zircaloy II. L. M. Osika. Knolls Atomic Power Lab. U. S. Atomic Energy Commission Pubn., KAPL-M-LMO-2, May 21, 1959,



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An X-ray diffraction analysis was applied to the measurement of a corrosion film on corrosion tested Zircaloy II. Coating thicknesses in the range of 10^{-5} to 10^{-2} cm can be determined. The method is generally applicable to coatings of any material where X-ray-absorption coefficients are known.—NSA. 18534

2.3.5, 3.8.4, 4.3.2

Electromotive Force Measurements for the Comparative Estimation of the Protective Properties of Natural (Oxide and Chloride) Films of Some Metals. (In Russian.) N. A. Zapol'skaya. J. Phys. Chem. U.S.S.R. (Zhur. Fiz. Khim.), **32,** No. 3, 569-572 (1958).

Zapol'skaya studied corrosion-resistance by the e.m.f. effect of iron, nickel, magnesium, aluminum, zinc, tin and silver in acid and alkaline solution, and gives theoretical explanations of the values obtained. Although aluminum has the same potential in 0.1 normal hydrochloric acid and 0.1 normal nitric acid solution, the e.m.f. effects are different in these two solutions, for in nitric acid solution the oxide film increases in thickness, while the chloride film in hydrochloric acid solution becomes more porous on prolonged exposure, permitting continuous corrosion by Cl^- ions.—MA. 17649

2.3.5, 2.4.2, 5.3.4

Electrical Method of Measuring Carburized Case Depths and Thickness of Non-magnetic Electrodeposited Coatings. (In Russian.) T. I. Pavlov. Zavodskaya Lab., **23,** No. 1, 67-69 (1957). Translation Available from: Henry Brutcher, Technical Translations, P. O. Box 157, Altadena, Calif.

Development of an electrical method for precise determination of depth of carburized cases on steel. Description of device used: principle, electronic voltmeter. Relationship of potential differences along bridge of device to carburized-case depth. Results obtained on plain carbon and low chromium steels proving that not only case depth, but quality of case as well is indicated. Calibration curves. Application of method without any alterations to measurement of thickness of nonmagnetic electrodeposits, giving even more accurate results. 5 figures.—HB. 17999

2.3.5, 3.2.2, 3.7.3

Determination of Susceptibility to Intergranular Corrosion of Welds in Structures. (In Russian.) N. D. Tomashov and O. G. Deryagina. Zavodskaya Lab. **23,** No. 6, 679-682 (1957). Translation available from Henry Brutcher, Technical Translations, P. O. Box 157, Altadena, Calif.

Description of a nondestructive test of steel for tendency to intergranular corrosion, especially useful in testing weldments. Authors' study of distribution of potentials across welds (the differences in potential being due to dissimilar heat-treating effects). Steel used for study: higher-carbon 18-8 titanium. Best electrolyte for testing at ordinary temperature. Set-up for potential measurements under a microdrop of electrolyte. Variation of electrode potentials with time, under drops of various solutions. Change in steady electrode potentials with heat treatment. Distribution of potentials on surface of welded 18-8 titanium steel measured in various solutions.—HB. 17893

2.3.6, 3.8.4

The Nature of Thin Oxidation Films on Metals. (In German.) Libuše Vlasáková. Werkstoffe u. Korrosion, **9,** No. 8/9, 536-538 (1958).

Films on aluminum, zinc and copper were examined by measurements of the elliptically polarized reflected light-beam from the surface and also by electron emission. With the first method curves for adsorption and desorption of air, oxygen and argon were obtained using aluminum. Electron-emission curves for various temperatures and times for zinc and copper show the difference between initial emission and that after a layer has formed. A relationship exists between passivation (of zinc) and electron emission.—MA. 17598

2.3.6, 5.9.4, 6.3.13

The Determination of the Thickness, Dielectric Constant, and Other Properties of Anodic Oxide Films on Tantalum from the Interference Colors. L. Young. Proc. Total Soc., A, **244,** No. 1236, 41-53 (1958) Feb.

Electropolished 99.9% tantalum sheet was anodized in 0.2 N-sulfuric acid solution and the properties of the oxide film formed were determined, using spectrophotometric measurements of the Å of minute specular reflectivity at 11° incidence to derive the film thickness. The following values were obtained: refractive index $= 2.14 + 0.292/(Å/10^3 A.-2.305)^{1.2} - 1\%$; $d = 7.93 \pm 0.25$ g/cc; effective dielectric constant $= 27.6 - 5\%$ at 1 kc/s; field strength in the oxide at current density of 9.55 m.amp/cm^2 $= 6.61 \times 10^6$ V/cm at 25.8 C., independent of thickness; ratio of true to apparent area $= 1.05$. 21 references.—MA.

2.3.6

Use of the Electron Microscope for Direct Examination of Thin Films of Metals and Alloys. Pts. I, II. (In French.) C. Beauvais. Métaux Corrosion-Inds., **34,** 247-257, 291-301 (1959). English Translation HW-tr-8. Available from John Crerar Library, 86 E. Randolph St., Chicago 1, Illinois, Attn: SLA Translation Center.

Methods for preparing thin films for examination by electron microscopes are described. Results are discussed and electron micrographs obtained by these methods with single and polyphase metals, and alloys are presented. It is pointed out that films of a great number of metals and alloys can be prepared in suitable form for examination by this method. A study based on electron microscopic methods but completed by other methods is included.—NSA. 19005

2.3.6, 3.7.4, 6.3.21

The Orientation-Dependence of Etching Effects on Germanium Crystals. (In English.) P. J. Holmes. Acta Metallurgica, **7,** No. 4, 283-290 (1959).

Polished hemispheres of single-crystal germanium were treated with a variety of etchants. Real etch features were distinguished by their recurrence in positions determined by the crystal symmetry. The hdrofluoric acid-H_2O_2 etchants showed certain effects only over angular ranges of a few degrees. There was evidence that the variations were due not only to etch-rate differences but also to changes in the actual mechanisms of etching with orientation. 13 references.—MA. 19213

2.3.7, 2.2.5, 5.4.5

Practical Problems in Paint and Lacquer Testing. (In German.) D. Wapler. Metall, **12,** No. 6, 512-515 (1958); See also: Metalloberfläche, **12,** No. 4, 113-119 (1958).

Test procedures for determining the mechanical and physical properties and service life of paint and lacquer coatings on metals are discussed.—MA. 18992

2.3.9

In Situ Tracers Project. Quarterly Progress Report No. 2 for June-August, 1959. William H. Johnston Labs., Inc. U. S. Atomic Energy Commission Pubn., AECU-4383, 32 pp. Available from Office of Technical Services, Washington, D. C.

Theoretical studies are reported on an evaluation of the potential usefulness of **in situ** tracers in industrial application of radioisotopes. Proposed maximum isotope concentrations for **in situ** tracers are tabulated for tritium, carbon-14, iodine-131, iodine-132, iron-59, magnesium-56, phosphorus-32, potassium-42, sodium-24, sulfur-35, yttrium-90, and zinc-65. Theoretical derivations were made to evaluate the required levels of tracer concentration as a function of instrument background, efficiency of detector, required statistical reliability and tracer nuclide. Possible areas where radioisotopes can be used in the paint industry were surveyed. As a result of conferences with representatives of the National Paint, Varnish and Lacquer Association it was decided to place emphasis on the development of tracer methods for the measurement of air pollution from the paint industry, studies on film deterioration, and measurements of film thickness of paints. Progress is reported on the design and construction of low-level soft beta counters and a large low-level liquid scintillation gamma counter for industrial use. Preliminary design drawings are included.—NSA. 19241

2.3.2, 2.3.7, 5.4.5

Laboratory Tests for Paint. R. E. Gackenbach. Am. Paint J., **43,** No. 33, 9-10 (1959).

Laboratory tests used to evaluate primers (one or two coats) and topcoat paints (one coat only) are described. The salt spray test may be replaced by a water bath test as it gives almost identical results and is easier to carry out. Evaluation is based on six characteristics: ease of application, drying time, dry film thickness of 1.5 mills minimum per coat, water resistance, scratch corrosion and chemical resistance. It is emphasized that laboratory tests only reveal which paints are unsuitable. They do not pick out the best paint.—RPI. 18266

2.3.5, 3.6.8

Oxygen-Evolution Reaction of Gold Anodes. Pt. I. Accuracy of Overpotential Measurements. Sidney Barnartt. J. Electrochem. Soc., **106,** 722-729 (1959) August.

Various sources of error in overpotential measurements are evaluated, a new cell design for uniform current distribution described, and the current density limits determined for measuring η to $\pm$ 1 mv.—BTR. 18253

2.3.5, 3.5.5, 5.3.2

An Electro-Chemical Method of Obtaining an Index of Porosity of Metal Coatings. M. Clarke and S. C. Britton. Trans. Inst. Metal Finishing, **36,** Pt. 2, 58-66 (1958/59) Winter.

If porous metal coating is treated anodically in such conditions that cur-

after 2 years of chemical spills and weather...

HYPALON® Tank Truck Finish Shows No Damage...Stops Corrosion

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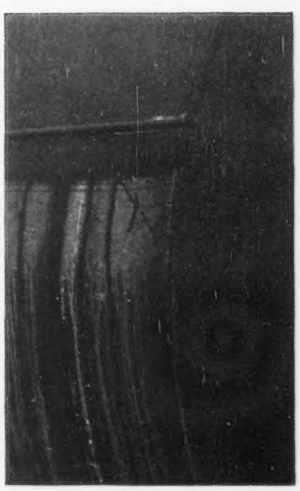
Ask your coatings supplier about HYPALON coatings or write for your free copy of PROTECTIVE LININGS AND COATINGS. This comprehensive brochure is designed for engineers with a corrosion problem. E. I. du Pont de Nemours & Co. (Inc.), Elastomer Chemicals Department C-8, Wilmington 98, Delaware.



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rent passes almost entirely via base metal exposed at pores and polarization of basis metal is insensitive to change of current, displacement of potential as current is varied should depend on electrical resistance of electrolyte in pore channels. Required conditions were found for coatings of tin, tin-nickel alloy, speculum, nickel and copper on steel. Plot of displacement of potential against applied current gives straight lines with slope depending on conductivity of electrolyte (3 percent sodium chloride). Resistance values have satisfactory relation to results of other porosity tests (sulfur dioxide porosity test) and are regarded as useful numerical index of porosity. Method can also be applied to coatings of tin-nickel alloy on copper or nickel. Tables, graphs, photographs of sulfur dioxide test specimens. —INCO. 18127

2.3.9, 8.4.5, 3.5.4

A Simple Graphical Method for Computing In-Reactor Corrosion Temperatures. R. L. Dillon. General Electric Co., Hanford Atomic Products Operation. U. S. Atomic Energy Commission Pubn., HW-55045, Feb. 19, 1958, 8 pp. Available from Office of Technical Services, U. S. Department of Commerce, Washington 25, D. C.

A graphical method is described for estimating the corrosion temperature for fuel elements under irradiation. (auth) —NSA. 18100

2.3.9, 2.4.3, 3.8.4

Comparing Marketing Methods in Investigations of the Mechanism of the Oxidation of Metals and Alloys. (In Polish.) Lucjan Czerski, Stanislaw Mrowec and Teodor Werber. Archiwum Hutnictwa, **3,** No. 1, 37-48 (1958).

Three methods were investigated: layer, microscopical and autoradiographical. The microscopic method is most convenient for exactness and simplicity. —BTR. 18130

2.3.9, 2.4.3

Large Replicas in Non-Destructive Metallography. (In French.) Pierre A. Jacquet. Mém. sci. Rev. Mét., **56,** No. 2, 101-110 (1959). See also Rev. Mét., **55,** 531-554 (1959) June.

Further applications of a portable electrolytic cell used for local polishing preparatory to obtaining a nitro-cellulose replica, are described. They include examination of failed stress-corrosion and fatigue samples and metallographic control of cast blanks. Detection of hair cracks in brass subjected to bending fatigue is facilitated since the oxide film, characteristic of the areas of greatest plastic deformation, does not interfere. 13 references.—MA. 19244

3. CHARACTERISTIC CORROSION PHENOMENA

3.2 Forms

3.2.2, 6.3.15, 3.7.2

The Effect of Composition of the Hydrogen Embrittlement of Alpha-Beta-Titanium Alloys. R. I. Jaffee and D. N. Williams. Trans. Amer. Soc. Metals, **51,** 820-841; disc., 841-842 (1959).

The hydrogen embrittlement of binary and ternary titanium alloys containing molbdenum, vanadium, aluminum, chromium, iron, manganese, niobium and tantalum decreased as the amount of β-phase and aluminum content increased, but became greater as the oxygen content increased. Molybdenum was the most effective β-stabilizer and iron and manganese the least. The combined effect of the β-stabilizers was additive. Tolerance to embrittlement can be improved by increasing solubility of hydrogen in β by addition of molybdenum, or solubility in α by addition of aluminum. A microstructure having a long, continuous interface between α and β promoted slow-strain embrittlement, but impact embrittlement occurred only when a hydride phase was observed. 10 references.—MA. 19239

3.2.2, 3.8.4

The Effect of Bubbling on Corrosion. (In Japanese.) Yahiko Kadono. Corrosion Engineering, **7,** No. 5, 30-34 (1958) Sept.

Localized corrosion which cannot be explained by conventional theories (i.e., galvanic corrosion, concentration cell, or erosion), is frequently found in petroleum refinery equipment.

This particular case of localized corrosion is caused by bubble formation. 1) Due to alternate contact with gas and liquid phases, distribution of energy (on metal surface) is not uniform and corrosion accelerated. 2) The bubbles in the liquid require energy in order to form. 3) Energy on metal surface varies with composition of gas, liquid and metal. 4) The name "alternate phase effect" is proposed for this new theory. 18921

3.2.2, 6.3.15

Hydrogen Contamination in Titanium and Titanium Alloys. Pt. V. Hydrogen Embrittlement. Period covered March, 1957 to March, 1958. D. N. Williams, F. R. Schwartzberg and R. I. Jaffee. Battelle Memorial Inst. U. S. Wright Air Development Center, Technical Rept. 54-616 (Pt. 5), November 24, 1958, 99 pp.

Extensive investigations intended to provide information relating to the mechanism of hydrogen embrittlement in titanium-phase alloys were carried out. Studies included detailed measurements of the effects of temperature, strain rate and hydrogen content on embrittlement, examination of all-alpha and all-beta alloys for susceptibility to embrittlement, calculations of probable hydrogen solubility in alpha and beta titanium, and several other informative investigations. An embrittlement mechanism is outlined. Alloys previously prepared and examined to determine the effects of alloy content and microstructure on hydrogen embrittlement were further studied by means of notched stress-rupture tests. This work resulted in alteration of previously expressed conclusions regarding alloying and microstructural effects. (auth)—NSA. 18983

3.2.3, 3.7.4, 6.3.11

A Possible Action of Air on Very Thin Silver Films. (In French.) Jean Trompette. Compt. rend., **246,** No. 23, 3235-3238 (1958).

On the basis of work by David (Z. Physik, **114,** 389, 1938), the assumption that air initiates a displacement of fine grains, which aggregate to form larger grains, in thin silver films confirms previous results by Trompette. (Compt. rend, **246,** 753, 1958).—MA. 18964

3.2.2, 3.8.3, 6.2.3, 3.4.8

Pitting Corrosion on Passive Electrodes of Unalloyed Steel in Sulfuric Acid Containing Chlorine-Ions. (In German.) Hans-Jurgen Engell and Nikolas D. Stolica. Arch. Eisenhuttenw., **30,** 239-248 (1959) April.

Steel electrodes are passivated in 1 N sulfuric acid at 25 C by a potential of +1750 mV. A lower potential is applied to the electrodes in 1 N sulfuric acid and in the presence of chlorine-ions. The increase of dissolution current at a constant electrode potential is due to local attacks on the electrode surface by chlorine-ions.—RML. 19101

3.2.2, 3.5.8, 6.2.5, 6.3.15

Hydrogen Embrittlement in Steels, Titanium Alloys, and Several Face-Centered Cubic Alloys. P. A. Blanchard, R. J. Quiqq, F. W. Schaller, E. A. Steigerwald and A. R. Troiano. Case Inst. of Tech. U. S. Wright Air Development Center, Technical Report 59-172, April, 1959, 129 pp.

This report, which describes the influence of hydrogen on the mechanical properties of high strength steel, titanium and face-centered cubic metals, is divided into three sections. **Delayed Failure in High Strength Steel.** The initiation of localized cracking in a hydrogenated high strength steel specimen was found to be dependent on the development of a critical hydrogen concentration and relatively insensitive to the magnitude of the applied stress. The stress played an essential role in the delayed failure process by providing the means for grouping the hydrogen. Assuming that the rate of stress-induced diffusion was a direct function of the applied stress, the predicted relationship between incubation time and stress agreed reasonably well with experimental data. **Hydrogen Embrittlement and Strain Aging In Titanium Alloys.** Low strain rate embrittlement in titanium alloys can be classified as a strain aging phenomenon. Prestraining and aging an alpha-beta titanium alloy resulted in a ductility minimum at some intermediate aging time. It appears that hydrogen migrates to a region of inhomogeneous strain, where a high stress state exists and creates this embrittlement. The restoration of ductility at long aging times was attributed to recovery with subsequent redistribution of hydrogen. Low strain rate hydrogen embrittlement was obtained for an alpha alloy and a beta alloy. Hydrogen in small quantities seemed to aid creep resistance in the alpha alloy. The beta alloy was resistant to nominal quantities of hydrogen (4200 ppm), but did show embrittlement at higher levels. **Hydrogen Embrittlement of Several Face-Centered Cubic Alloys.** The hydrogen embrittlement of austenitic nickel-chromium-iron alloys and OFHC copper has been investigated. Nickel-chromium-iron alloys were embrittled by hydrogen and their embrittlement was demonstrated to be of the same nature as that of steel. A qualitative mechanism was presented which indicated that only the transition metals should be capable of conventional hydrogen embrittlement. This mechanism also accounted for the observed decrease of embrittlement in the austenitic nickel-chromium-iron alloys with increasing (iron plus chromium) content. (auth)—NSA. 19047

3.2.2, 5.9.4

Investigation of the Effect of Hydrogen Embrittlement and Its Relief in Phosphate Coated AISI 4340 and 1095 Steels. P. H. Austin, S. V. Chek and Wm. Lurie. Naval Weapons Plant, Washington, D. C. NWPW-T-20-59; NAVORD rept. no. 6509, August 12,

1959, 33 pp. Microfilms and Photocopies available from Library of Congress, Photoduplication Service, Publications Board Project, Washington 25, D. C.

The effect of hydrogen embrittlement resulting from the phosphate coating of AISI 4340 and 1095 steels, at various hardness levels, was investigated by the use of sustained load (ring) tests made at 80 per cent of the uncoated notch breaking strength of the steels for corresponding hardnesses. 5 references.—GRR. 19028

3.2.3, 6.3.20

Nature and Properties of the Thin Oxide Film Formed on Zirconium and Zircaloy. E. A. Gulbranson and K. F. Andrew. pp. 465-476 of "Reactive Metals." Metallurgical Society Conferences, Vol. 2, 1959. Interscience Publishers, Inc., New York 1, N. Y.

Hydrogen permeation suggested as a method for study of protective oxide films forming on zirconium and Zircaloy-2. Data for hydrogen reactions on metals with various surface preparations and pretreatments at 150 C test temperature and 2.4 cm mercury of hydrogen pressure. 10 references.—RML. 19170

3.3 Biological Effects

3.3.1, 4.6.11, 3.8.4

Biological Factors Influencing Sea Water Corrosion. (In French.) Bernard Callame. Corrosion et Anticorrosion, **7,** 173-177 (1959) May.

Oxygen enrichment and evaluation of the pH-number by the photosynthesis of green plants. Reduction of oxygen content and of the pH-number by animal metabolism. Formation of hydrogen sulfide and carbon dioxide and hydrogen consumption by anaerobes.—RML. 18346

3.3.2, 5.4.8

Susceptibility of Matrix Constituents of Antifouling Paints to Microbial Attack in Sea Water. ROBERT L. STARKEY. *Can. J. Microbiology,* **3,** Suppl., 231-238 (1957) March.

Determinations of oxygen consumption as an index of microbial attack and of weight losses were both similar ,and reflected the rate of decomposition. Ester gum and Pentalyn compounds did not decompose.—BTR. 14376

3.3.4

How to Detect and Control Corrosion-Causing Bacteria. C. H. Oppenheimer. Univ. Texas. World Oil, **147,** No. 7, 144-147 (1958) Dec.

Discussion of methods of detecting bacterial corrosion. The first action to prevent bacterial corrosion should be taken before iron or steel equipment is installed. Type of control to be used will be influenced by environmental conditions such as water content or soil drainage, presence of water spray, amount of electrolytes, presence of organic matter, acidity or alkalinity and oxygen content. Bacterial corrosion can be controlled in certain situations by application of protective coatings, use of cathodic protection and application of chemical biocides to kill living bacteria. 30 references.—INCO. 17532

3.4 Chemical Effects

3.4.3, 3.4.7

The Value of pH Established by Hardly Soluble Metals Corrosion Products. (In Russian.) N. P. Zhuk. J. Phys. Chem. (Zhur. Fiz. Khim.), **32,** No. 12, 2754-2760 (1958).

A comparison was made between pH calculated for the almost insoluble corrosion products of aluminum, cadmium, copper, iron, magnesium, manganese, nickel, lead, tin, zinc (oxides and hydrated oxides), and pH obtained in practice in 0.1 M-sodium sulfate solution, it being assumed that this solution does not react with the corrosion products causing changes in pH. The initial pH values were 5, 7, 9 and 4, 7, 10 in two series of experiments. During corrosion of each metal the pH tends to level off at a rate of $\propto$ the corrosion rate of the metal and inversely $\propto$ the difference of the initial pH values. The pH values obtained experimentally from the corrosion products agree closely with those calculated from the solubility products of these compounds. 14 references.—MA. 19029

3.3.4, 1.3

The General Physiology of the Sulfate Reducing Bacteria in Relation to Corrosion. Pt. II. Robert L. Starkey. Producers Monthly, **22,** No. 8, 12 + 12 pp. (1958) June.

A review with 80 references. To be continued.—BTR. 16632

3.4.8, 6.3.10, 3.7.2

Investigation of Sulphur Attack on Nickel and Nickel Alloys. (In German.) Irmtraud Pfeiffer. Z. Metallkunde, **49,** 267-275 (1958) May.

Kinetics of sulfurizing reaction at 620. Rate of sulfide formation is dependent on alloying additions. Microscopic study of sulfide layers; intercrystalline corrosion.—MR. 16588

3.4.9, 6.3.21

Autoclave Testing of Mechanically Jacketed Thorium Slugs in Water. Frank L. Yaggee. Argonne National Lab. U. S. Atomic Energy Commission Pubn., ANL-4973, Jan. 29, 1953 (Declassified Feb. 12, 1957), 19 pp. Available from Office of Technical Services, Washington, D. C.

Water corrosion tests on mechanically jacketed and pin-holed thorium slugs show that these slugs fail in a manner similar to that observed for mechanically jacketed and tested uranium slugs. The proposed mechanism for the water corrosion of these jacketed slugs is analogous to the water corrosion mechanism of jacketed uranium slugs presented in the project literature. A bare thorium slug appeared to be more resistant to corrosion by water than a mechanically jacketed slug during the first half of the autoclave test. After approximately 90 hours of testing both the bare and the mechanically jacketed thorium slugs were severely corroded by water. (auth). —NSA. 16413

3.5 Physical and Mechanical Effects

3.5.3

Hydromechanical Aspects of Cavitation Erosion. (In Russian.) K. K. Shal'nev. Izvest. Akad. Nauk SSSR, 1958, (Tekhn.), No. 1, 52-62.

Work on erosion is summarized. The influence of several factors on erosion rate was studied. An investigation of the various stages of erosion showed there was a maximum rate of erosion at a given length of cavitation, depending on the experimental conditions. Linear relationships exist between the rate of flow and the intensity of erosion, and Reynolds No. and the intensity of erosion. 24 references.—MA. 18950

3.5.4, 6.6.4, 3.7.4

Radiation Effects on the Oxidation Rate and on Other Chemical Properties of Graphite. G. R. Hennig, G. J. Dienes and W. Kosiba. Paper prepared for 2nd United Nations International Conference on the Peaceful Uses of Atomic Energy, 1958, 12 pp. A/CONF.15/P/1778. Available from Office of Technical Services, Washington 25, D. C.

Properties of graphite are altered by irradiation with fast particles because these produce various lattice defects such as vacancies, interstitial atoms, ions, clusters of both and possibly dislocations. 10 references.—MR. 19192

3.5.4, 6.3.6, 6.4.2, 6.2.5

Corrosion Behavior of Structural Metals in Ionized Air. I. V. Shatalov and V. A. Nikitina. Paper presented at 2nd International Conference Peaceful Uses of Atomic Energy, Geneva, 1958, A/CONF.15/P/2042, 19 pp. On Deposit at the Depository Libraries, Available in Microcard form from the Microcard Foundation, 901 26th St., N.W., Washington 7, D. C.

Corrosion of copper, aluminum and carbon and stainless steels in air ionized by α radiation was studied. Moist and dry air were used. Corrosion resistance decreased with humidity and radiation intensity. Corrosion products were generally nitrates.—MA. 18944

3.5.4, 3.7.3, 6.2.5, 4.7

Cyclotron Irradiation of Type 347 Stainless Steel Welds in NaK at Elevated Temperatures. A. A. Epp, Jr., R. D. Moeller and H. Pearlman. North Am. Aviation, Inc. U. S. Atomic Energy Commission Pubn., North American Aviation, Special Rept. No. 74 (Del.), Jan. 7, 1953 (Declassified with Deletions April 11, 1957), 42 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington 25, D. C.

Capsules of Type 347 stainless steel, containing samples of arc welded 347 immersed in sodium-potassium of 78 wt.% potassium were irradiated with deuterons on the 60 in. cyclotron at Berkeley while maintained near 450 C. Power densities ranging from 2500 to 5000 watts/cm^3 were achieved in the weld samples near the sodium-potassium interface. The irradiations were between 10 and 20 hrs in length, at average beam currents from 1.5 to 3 micro-amperes. Control runs in which the capsules were heated, but not irradiated, and in which a weld sample was irradiated in an evacuated capsule, were also made. No conclusive evidence was obtained that weld corrosion was accelerated in the irradiated runs, compared with unirradiated controls. In addition to weighing the samples, examining them metallographically for evidence of corrosion and analyzing the sodium-potassium chemically after the runs, observations were also made of surface hardness, grain size and x-ray-diffraction patterns of the weld samples. No significant differences were found between the irradiated and control samples. (auth)—NSA. 17875

3.5.8, 3.6.5

Effect of Stress on the Corrosion Potential of Steel. Pt. I. Tensional Stress. (In English.) Eino Uusitalo. Suomen Kemistilehti, **31,** No. 12, 395-399 (1958).

In the elastic range stretching produces a temporary shift of the potential in the anodic direction. At the plastic limit the potential first undergoes a large shift in the anodic direction but then changes rapidly in the reverse direction. 11 references.—RML. 17610

3.5.8, 6.3.6

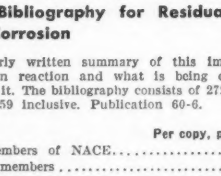
The Initiation and Propagation of Cracks in the Stress Corrosion of Alpha-Brass and Similar Alloys. A. J. Forty. pp. 99-120 of "Physical Metallurgy of Stress Corrosion Fracture." Metallurgical Society Conference, Vol. 4, 1959. Interscience Publishers, Inc., New York 1, N. Y.

Theory of stress corrosion cracking in metals taking into account the combined action of stress and chemical environment. Special attention is paid to the alpha brass/ammonia system. 16 references.—RML. 19161

3.5.8, 6.2.5

Metallographic and Crystallographic Examination of Stress Corrosion Cracks in Austenitic Chromium-Nickel Steels. J. G. Hines and R. W. Hugill. pp. 193-226 of "Physical Metallurgy of Stress Corrosion Fracture." Metallurgical Society Conferences, Vol. 4, 1959. Interscience Publishers, Inc., New York 1, New York.

Stress corrosion cracks produced in 0.318-0.635-cm. diameter specimens under 42 per cent magnesium chloride solution under axial tensile stress. Effects of stress severity and austenite and martensite flakes on stress corrosion cracks; effects of stress distribution, gamma ferrite grains and crystallographic planes on the crack paths. 18 references.—RML. 19219



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3.5.9, 3.5.8, 3.2.2, 6.2.4

Effect of Temperature on the Static Fatigue Characteristics of Hydrogen Embrittled 4340 Steel. E. A. Steigerwald, F. W. Schaller and A. R. Troiano. Case Inst. of Technology. U. S. Wright Air Development Center, Technical Report 58-178, April, 1958, 25 pp.

The effect of testing temperatures below room temperature on the static fatigue parameters of a hydrogenated high-strength steel was investigated. The results indicated that the incubation time required for the formation of the first crack in a specimen tested in static fatigue was controlled by the diffusion of hydrogen. The activation energy for this process was 7610 cal/mole. As the test temperature was lowered, the incubation time occupied a greater percentage of the total time to fracture until at —50 F the incubation and fracture times were essentially coincident. The kinetics of crack propagation at subzero temperatures indicated that cracking progressed in a discontinuous fashion. The delayed failure process involves a series of crack initiations rather than the continuous growth of a single crack. (auth)—NSA. 19001

3.6 Electrochemical Effects

3.6.5

Electrochemical Behavior of the Palladium-Hydrogen System. Pt. I. Potential Determining Mechanisms. Sigmund Schuldiner, Gilbert W. Castellan and James P. Hoare. J. Chem. Physics, **28,** No. 1, 16-19 (1958).

The potential of saturated α-palladium in hydrogen-stirred solutions compared with a Pt H electrode in the same solution is 0.0495 ± 0.0005 V. The potential-determining reaction of α-palladium appears to be independent of hydrogen pressure. Pure palladium spontaneously absorbs hydrogen in hydrogen-stirred solution until the saturation limit (H:Pd = 0.025 ± 0.005) of the α-phase is reached. Between the atomic ratios of H:Pd = 0.03 and 0.36 both the α and the β phases co-exist and the mixed potential is determined by that of the α-domains. Over the range H:Pd = 0.36—0.6 the potential is a function of the hydrogen-content of the palladium. 13 references.—MA. 17700

3.6.5

The Potential of Metal Surfaces at Oxidation and Adsorption. W. Schaaf. Z. Angew. Phys., **10.** No. 11, 503-511 (1958).

Potential measurements of metallic surfaces show that the potential of coated metal is not a simple function of coating thickness but goes through a maximum and minimum. The oxidation of iron and coper wire, and small plane surfaces of copper, iron, nickel, aluminum and silver, were investigated. The potential of the anodic oxide coating on aluminum goes through a sharp maximum at high coating thickness. The adsorption-desorption time dependence of stearic acid on highly polished tungsten was investigated by potential measurement.—EL. 19025

3.6.8, 3.8.3, 6.3.11, 6.2.2

Investigation of Phase-Boundary Impedance for Anodically Loaded Platinum, Indium and Iron Electrodes. K. Franke, C. A. Knorr and M. Breiter. Z. Elektrochem., **63,** No. 2, 226-238; disc., 238 (1959).

Phase boundary impedance was measured for platinum and iridium electrodes in 0.6 N-sulfuric acid and iron in 0.1 N-sodium hydroxide bathed in hydrogen using anodic d.c. polarization of 0-2000 mV and a.c. 5-40 kc/s; stationary current/hydrogen voltage curves were also obtained. For platinum, oxygen imposes a boundary value on the capacity, but is not reactive enough to cause pseudo-capacity as found for hydrogen adsorption. For iridium large pseudo-capacity is caused by adsorbed oxygen. Iron is similar to platinum for the capacitive component, but the ohmic component is greater. Both components show a big change in value and frequency-dependence as the electrode changes from the active to the passive state. 32 references.—MA. 19147

3.6.8, 3.8.4

Feedback Theory of Hydrogen Overvoltage. J. F. Chittum. Nature, **183,** No. 4661, 589-590 (1959). Cf. Conway and Bockris, ibid., **178,** 488, 1956.

A metallic-bonded hydrogen-metal complex exists on the surface where hydrogen is evolved. Decreased hydrogen-adsorption energy with increased surface coverage may be due to residual covalent metallic bonding by s-electrons. Metals are divided into three groups according to the rate-determining step in hydrogen evolution: $H_3O^+(M) + e^-(M) = H(M) + H_2O$ for mercury, thallium and lead; $\frac{1}{2}H_2(M) = H(M)$ for cadmium, aluminum, copper, gallium, iron, silver, etc.; $2H(M) = H_2(M)$ for platinum, palladium and rhodium. 14 references.—MA. 19075

3.6.8, 6.2.2, 5.3.4, 4.3.5

The Effect of Diffusing Atomic Hydrogen on the Hydrogen Overvoltage of Iron and Iron Protected by Electrodeposited Coatings in Acid and Alkaline

Solutions. (In Russian.) A. I. Oshe and I. A. Bagotskaya. J. Phys. Chem. USSR (Zhur. Fiz. Khim.), **32,** No. 6, 1379-1388 (1958).

Diffusing atomic hydrogen increases the hydrogen overvoltage (η) of electrodeposited nickel in acid and alkaline solutions at low values of η, but with increase in polarization the intensity of the effect decreases, and eventually η begins to decrease. On electrodeposited copper in alkaline solution η shows an increasing tendency only. The decrease of η at constant polarization with increase of the quantity of diffusing hydrogen grows at first and then approaches the limiting value in accordance with theory. The same phenomenon occurs at increased polarization and constant i_1/i. All the effects of decreasing η at high values of η considered here are higher than theoretical.—MA. 18907

3.6.9

Intermediate Analysis of Stray Current Corrosion. Ted Watson. West Virginia University, Technical Bulletin No. 52, 72-90 (1958) October.

The analysis of stray DC currents in the ground, their effects on buried structures and the measures taken to relieve the buried structures from their harmful effects.—BTR. 18998

3.6.5, 3.6.6

Electrode Potentials and Corrosion. W. F. Higgins. Corrosion Technology, 6, No. 10, 313-314 (1959) Oct.

Clarifies much of the misunderstanding that exists about relationship between tables of normal electrode potentials and galvanic series of metals on one hand, and possibilities of galvanic or bimetallic corrosion on the other. Some practical examples are given for cadmium-magnesium, zinc-magnesium alloy-aluminum alloy, silver-silver, copper-steel, zinc-steel and zinc-steel couples.—INCO. 19216

3.6.5

Electromotive Force Series of Metals in Fused Salts and Activties of Metal Chlorides in 1:1 Molar KCl-NaCl Solutions. S. N. Flengas and T. R. Ingraham J. Electrochem. Soc., **106,** 714-721 (1959) Aug.

The potentials of a series of metal-metal chloride systems in an equimolar mixture of molten potassium and sodium chlorides have been measured against a silver-silver chloride reference electrode. An electromotive force series of metals at different temperatures has been established for this particular solvent. Experiments with a chlorine electrode have shown that the solutions of silver chloride in the potassium chloride-sodium chloride solvent are ideal, and hence, deviations from ideality of the other chlorides in this solvent can be attributed directly to the formation of complexes in the melts. Three types of behavior have been observed for the salts studied in this solvent: ideality, and positive and negative deviations from ideality. The theoretical aspects of complex formation in melts are discussed. 19148

3.6.5, 3.6.6

Galvanic Series of Metals in Respect of Corrosives Encountered in Practice. (In German.) J. Elze. Berlin-Dahlem. Werkstoffe u. Korrosion, **10,** No. 12 (Suppl.), 737-739 (1959) Dec.

The well known electromotive series of elements can only to a limited extent be regarded as a criterion for the corrosion of metals. An effort has therefore been made to obtain, for fresh water and sea water, "practical" values of a galvanic series by means of measurements. It was found, however, that the open circuit potential cannot serve as the sole criterion for the corrosion performance of metal combinations. It is also essential to know the slope of the current/voltage curve of the electrode in the corrosion agent. 19117

3.7 Metallurgical Effects

3.7.2, 6.4.2

Effect of Small Quantities of Magnesium, Copper, Iron, Silicon and Surface Condition on the Resistance of Aluminum to Corrosion. (In Japanese.) T. Amitani. Sumitomo Metals, **11,** No. 1, 51-64 (1959) Jan.

Effects of small quantities of magnesium, copper, iron, silicon and surface condition on the resistance of aluminum to corrosion were investigated. Weight loss, decrease of mechanical properties, aspect of corrosion pit and Mylius test were considered.

(1) The addition of more than 0.1 percent of copper reduced the corrosion resistance of aluminum. (2) Addition of magnesium, even in very small amounts, improved the corrosion resistance of pure aluminum. Corrosion pit and Mylius tests showed improvement in corrosion resistance with addition of only 0.06 percent magnesium. (3) Addition of magnesium to aluminum contaminated with small amounts of copper did not improve the corrosion resistance and was rather harmful. (4) As the iron content increased, the area of corrosion pit was enlarged while the number of corrosion pits decreased.—JSPS. 18376

3.7.3, 3.7.4, 6.2.3

On the Corrosion of Welded Parts of Mildsteel. Preventive Effects of Various Other Deposited Metals of Welded Parts of Mildsteel. Kazuo Nishimaki and Hiroharu Uchiyama. J. Metal Finishing Soc. Japan, **9,** No. 4, 14-19 (1959) Apr.

Eighty specimens of different microstructure were prepared from arc-welded steel plates, which were welded with mildsteel, 1.5% chromium-molybdenum steel, 5% chromium steel, 13% chromium steel and 18 chromium-8 nickel steel deposited metals, under the adequate welding conditions. The mechanical properties of specimens and their defects were inspected by means of X-ray and supersonic waves.

Loss by corrosion, volume of hydrogen produced, and electrode potentials in 10% sulfuric acid, hydrochloric acid at the welded and annealed states, were measured. Welded strain was also studied.

1. The corrosion loss, and the volume of fuming hydrogen vary considerably with the microstructure.

2. Welding with 1.5% chromium-molybdenum steel is not effective in preventing corrosion.

3. Corrosion resistance of bead part is increased when 18-8 steel is used as the deposited metal.

4. The corrosion resistance of high sulfur mild steel plate is low as compared with low sulfur mild steel plate. 17507

3.7.3, 6.3.20, 8.4.5

Corrosion of Zircaloy-2 Sheet Weldments. R. Stein and R. E. Monroe. Battelle Memorial Inst. Welding J., **37,** No. 10, 468s-472s (1958) October.

Studies of fabrication and welding procedures for reactor fuel elements consisting of uranium dioxide plates jacketed in Zircaloy-2 sheet were conducted. Emphasis was placed on determining the suitability of economic welding techniques for joining Zircaloy-2. Comparison of bend ductility and corrosion properties showed no differences in weldments made in air with inert-gas shielding and weldments made in an inert-gas-filled vacuum-purged dry box. Weight gains of all corrosion samples were about the same. Fabrication, property and procedure data are reported. (auth)—NSA. 17849

3.7.3, 3.4.6

Controlled-Atmosphere Arc Welding. N. E. Weare and R. E. Monroe. Battelle Memorial Inst. Welding J., **37,** 1169-1175 (1958) Dec.

Many of the new engineering metals coming into widespread use in welding applications are sensitive to contamination by gaseous impurities such as oxygen, nitrogen or hydrogen. These metals and alloys may become embrittled or less resistant to corrosion because of gaseous impurities picked up during welding. Carefully controlled welding atmospheres are required to prevent harmful contamination of metals such as zirconium, titanium, molybdenum, hafnium, niobium and tantalum. The design, construction and operation of various types of equipment for preventing such contamination are discussed. Controlled-atmosphere equipment may range from large vacuum-purged dry boxes to simple trailing shields for use with

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standard welding torches. Shielding equipment for open-air use includes cups, trailing and leading shields, dragging shields and gas backups. Closed-atmosphere equipment discussed includes both flow-purged and vacuum-purged enclosures. (auth)—NSA.
17820

3.7.2, 6.2.5, 4.3.2

Influence of Addition Elements on the Corrosion Resistance of Chromium-Nickel-Molybdenum-Copper Steels in Sulfuric Acid Solutions. (In Russian.) E. V. Zotova. Khim. Prom. (Chemical Industry), 239-243 (1958). French Translation CEA-tr-R-696, on deposit at the Depository Libraries.

A study was made of the effects of the alloying elements chromium (5 to 27 percent), nickel (9 to 28 percent), molybdenum (0 to 3 percent), copper (0 to 3 percent), titanium (up to 0.5 percent), niobium (up to 1.5 percent) and carbon (0.02 to 0.3 percent) on the corrosion resistance of steels in 5 to 90 percent sulfuric acid at 80 C and in 5 to 50 percent boiling sulfuric acid. The results showed that chromium, nickel, molybdenum and copper improve the corrosion resistance to a great degree, but titanium, niobium and carbon have no effect. The conditions under which the metals improve the corrosion resistance are given. The steels having 5 to 9 percent chromium and 15 percent nickel display a fissuring corrosion in 5 to 60 percent sulfuric acid. The austenite-martensite two-phase steels have a considerably better corrosion resistance in 30 percent acid at 80 C than the single-phase austenite steel.—NSA. 19013

3.7.3, 3.2.2, 6.4.2

Burning Phenomena and Quality in Light-Alloy Welds. (In French.) R. Develay, M. Croutzeilles and R. Chevigny. Rev. Mét., **56**, No. 5, 409-417; disc., 417 (1959).

A suggested cause of blistering near weld beads in aluminum-magnesium alloys is oxidation of incipiently fused metal, which, because of its lower d, rises to the surface through the protective oxide layer. Blistering was reproducibly obtained by electrical-resistance heating of A-G5, A-U4G and A-Z8GU alloys. Particularly severe blistering occurred in aluminum-iron 0.4-Si 1.2-Mg 0.95 per cent alloy and is attributed to the high silicon content. This effect can be reduced or eliminated by homogenizing treatments for periods up to 48 hrs. at temperatures up to 500 C; by addition of $\lesssim$ 0.014 percent beryllium to A-G5 with 0.08 percent silicon, or $\lesssim$ 0.10 percent beryllium to A-SG; or most practicably, by using nitrogen or argon protective atmospheres. Flux covering, while suppressing blistering and giving a satisfactory superficial appearance, did not prevent intergranular oxidation.—MA. 19065

3.7.4, 6.2.2

Experimental Evidence of Boundary Migration. (In English.) A. Ferro, C. Sari and G. Venturello. Acta Metallurgica, **7**, No. 6, 429-431 (1959).

A letter. By heating pure iron and iron-silicon alloys in atmospheres slightly oxidizing towards silicon (pH_2O/pH_2 in the ratios 1.3×10^{-3}-4.3×10^{-3}) at 1000-1300 C, traces of the grain-boundary movements were revealed, owing to the recrystallized regions being less susceptible to oxidation than the unrecrystallized regions.—MA. 19124

3.7.4, 6.2.2

Structure of Ferrite Grain Boundaries. (In Russian.) V. N. Gridnev. Metalloved Term. Obrabotka Metallov, No. 1, 19-25 (1959) Jan. Translation available from Henry Brutcher, Technical Translations, P. O. Box 157, Altadena, California.

Difficulties obstructing the study of structural and concentration changes in grain boundaries. Etchant used for developing ferrite boundary layer. Formation and decomposition of boundary layer in technical iron (0.1 percent carbon) and in various alloyed irons (nickel, manganese, chromium, silicon, molybdenum, niobium, phosphorus; carbon: 0.02 percent max). Nature of boundary layer. Relation between boundary layer and mechanical properties of alloys.—HB. 19179

3.8 Miscellaneous Principles

3.8.2, 3.8.4

Sacrificial Corrosion and the Relative Corrodibility of Metals. G. Fitzgerald Lee. Corrosion Technology, **6**, No. 8, 245-247 (1959) Aug.

Discusses various aspects of sacrificial corrosion. Reference is made to the relationship which exists between corrosion and electrolysis and consideration given to considerable development of knowledge in this field since the war. Relationship existing between atomic number, atomic weight and corrodibility is explained. Tables show electromotive series, galvanic series and inherent corrodibilities.—INCO. 19134

3.8.2, 6.3.11

Electrochemical Mechanisms of Noble-Metal Hydrogen Systems. Part III. Electronic Configuration and Catalytic Activity. S. Schuldiner and J. P. Hoare. Naval Research Lab., Washington, D. C. NRL-5171, Project NR-475-000, June 19, 1958, 13 pp.

This report is the third of a series reviewing the experimental and theoretical findings of the electrochemical mechanisms of noble-metal/hydrogen systems. Relations between electronic configuration, the heat of adsorption of hydrogen atoms, catalytic activity, and the rate-determining step were experimentally demonstrated. It was shown that when the rate-determining step is an atomic desorption, the lower the atomic heat of adsorption, the higher the catalytic activity. When the slow discharge step is rate determining, the higher atomic heat of adsorption, the higher the catalytic activity. A linear relation between the number of positive holes in the d-band of nickel-palladium-hydrogen alloys and their catalytic activity was shown. The systems studied are α and β palladium-hydrogen, nickel-palladium-hydrogen and gold-palladium-hydrogen alloys. (auth)—NSA. 18947

3.8.2, 2.3.5

A Method Permitting Study of Electrode Exchange Current. (In French.) (Mme) Anne-Marie Baticle and Yves Thouvenin. Compt. rend., **248**, No. 6, 794-796 (1959).

From Nernst's law and anodic and cathodic diffusion-current considerations, curves showing the variation of log. apparent exchange current with electrode potential are constructed. The curves are utilized and discussed with reference to the study, around equilibrium, of ionic or electronic transfer for diffusion associated with, for instance, redox systems or corroded amalgam electrodes.—MA. 19027

3.8.2, 3.6.8, 6.3.11

Capacity of the Electrical Double Layer and Adsorption at Polarized Platinum Electrodes. Norman Hackerman and Pranjivan V. Popat. University of Texas. March 15, 1958, 31 pp. Microfilms and Photocopies Available from Library of Congress, Photoduplication Service, Publications Board Project, Washington 25, D. C. (PB 139197).

The differential capacity of the electrical double layer at polarized platinum electrodes has been investigated as a function of the type and concentration of anions.—GRR. 19190

3.8.2, 3.6.8, 3.8.3

Research on Anodic and Cathodic Behavior of Metals. Roberto Piontelli. Politecnico di Milano, Italy. Final Rept. on Contract AF 61(514)733-C. May, 1958, 30 pp. Microfilms and photocopies available from Library of Congress, Photoduplication Service, Washington, D. C. (PB 138877).

Content: Introduction; experimental methods; anode and cathode overvoltages of polycrystalline; gold, silver, copper, tin, lead, thallium, zinc, cadmium, indium, nickel, magnesium, titanium and iron; discussion on some general aspects of the metal electrode behavior; anion influence; passivation and passivity; systematic work of the electrochemical behavior of metallic single crystals; systematic work on the electrode behavior of metals in fused salts and especially of aluminum in cryolite-alumina baths; general conclusions and bibliography.—GRR. 18919

3.8.3, 3.5.9, 3.6.8, 6.3.10

Effect of Temperature on the Passivity of Nickel. (In English.) G. O. Okamoto, Haruo Kobayashi, Masaichi Nagayama and Norio Sato. Z. Elecktrochem., **62,** No. 6/7, 775-782 (1958).

Polarization curves for nickel in Normal-sulfuric acid were measured by a potentiostatic method and also by electrode impedance in the range 0.2-10 k.c./s. Two regions were found in the potential/current curve, where current is constant irrespective of potential, and where it increases steeply with potential. A break in the linear dependence of the log solution current/temperature relation is found at 60 C. This critical temperature is close to the temperature at which "exo-electron emission" takes place. 16 references.—MA. 18915

3.8.4, 6.3.17, 3.5.9

Oxidation of Uranium in Air at High Temperatures. R. K. Hilliard. General Electric Co., Hanford Atomic Products Operation, U. S. Atomic Energy Commission Pubn., HW-58022, December 10, 1958, 31 pp. Available from Office of Technical Services, U. S. Dept. of Commerce, Washington, D. C.

Reaction between metallic uranium and air was investigated at temperatures ranging from 300 to 1440 C. The effect on oxidation rate of time, furnace temperature, area/weight ratio and air flow rate was determined and the results presented graphically. The extent of self-heating due to the exothermic reaction was measured by recording both specimen and furnace temperatures. The time required for complete oxidation of cylindrical specimens was estimated and correlated as a function of furnace temperature and area/weight ratio. (auth) —NSA. 18167

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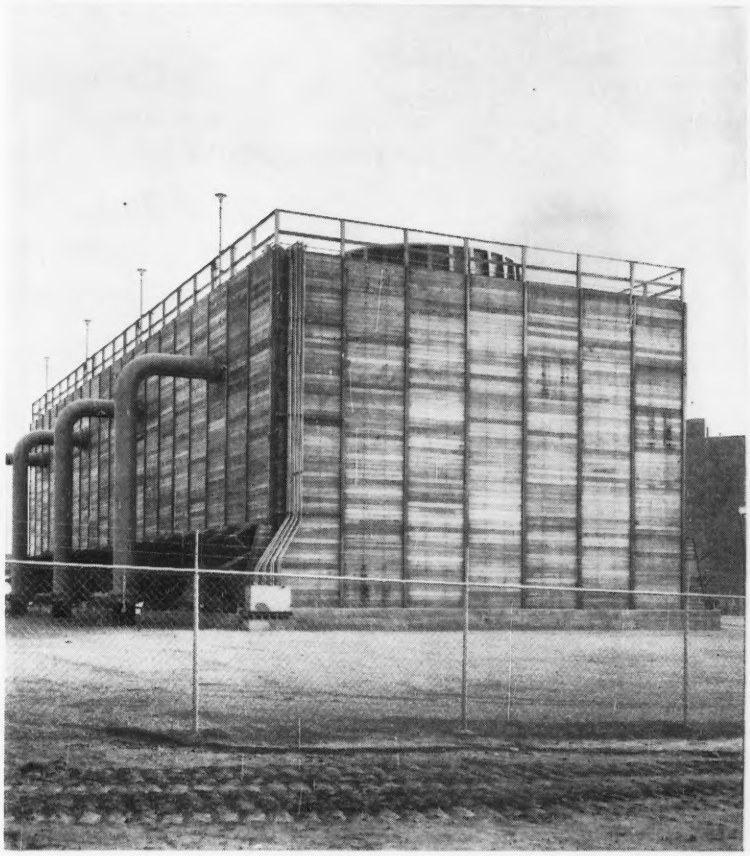

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(b) To provide forums and media through which experiences with corrosion and its prevention may be reported, discussed and published for the common good.

(c) To encourage special study and research to determine the fundamental causes of corrosion, and to develop new or improved techniques for its prevention.

(d) To correlate study and research on corrosion problems among technical associations to reduce duplication and increase efficiency.

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Materials Problems Associated With Uncooled Rocket Nozzles*

By JESSE D. WALTON, JR. and C. R. MASON, JR.

Abstract

A brief discussion is given of thermal shock, erosion, and catastrophic corrosion problems connected with uncooled rocket nozzles used with solid fuels. This is followed by a description of materials and methods used to meet these problems. A small oxygen-hydrogen rocket motor was used to screen materials.

Slip cast fused silica was used as a base material and erosion and corrosion resistance was provided in the throat area with arc-sprayed coatings or shells. Fabrication and evaluation techniques used are described as are forming methods and test results. 7.2

Introduction

THE RAPID technical advances which have been made in the fields of rockets, missiles and re-entry vehicles have brought with them numerous materials problems. Not the least of these are the ones associated with the development of materials for uncooled rocket nozzles. These problems have developed largely as a result of the increasing specific impulse being obtained with solid propellants. Materials for liquid propellant nozzles may be cooled by the fuel itself through regenerative processes. On the other hand solid propellant rocket nozzles must withstand the environment of the combustion products without cooling.

The conditions which might cause uncooled rocket nozzles to fail when used with high temperature high specific impulse solid propellants may be listed generally as follows: mechanical shock, thermal shock, erosion and corrosion.

Erosion may be considered to result from thermal or mechanical action. Melting or ablation would be examples of erosion resulting from the thermal environment. Corrosion might be considered to result from the following mechanisms: oxidation, reduction and/or chemical attack (fluxing). Oxidation could result from the presence of excess oxidizer. Reduction could result from the presence of unburned fuel either in the form of carbon or carbonaceous materials or from unoxidized ballistic modifiers such as aluminum. Chemical attack or fluxing would be more serious for ceramic type materials where boron or boron oxide, fluorine or fluorides, or oxides such as magnesium oxide and aluminum oxide could react with the ceramic oxide to lower its melting point.

The complete story of the combustion of solid propellants and the exact composition of the combustion products present in the exhaust stream of a burning solid propellant is unknown. It is conceivable that all of the above conditions are present during some period of time, or at some point in the exhaust stream. The variable conditions of combustion products at high velocities in the gas stream at the high temperatures presently being encountered may be considered an exceptionally severe environment in which to expect materials to exist. In addition, the heat flux presented by such a combustion products stream is extremely high and causes the materials to be raised to the flame temperature of the propellant almost instantaneously. Thus, there is an extremely short period of time over which a nozzle may act as a heat sink. However, when such short burning times are to be considered, the heat capacity and thermal conductivity of the nozzle are important.

It can be seen from this brief introduction into the problems associated with uncooled rocket nozzle materials that one single mechanism or cause of nozzle failuse is virtually impossible to isolate or consider alone. Thus, nozzle failure must be attributed to a combination of two or perhaps all of the above listed causes.

Graphite, for example, has long been used as a solid propellant rocket nozzle material. However, the poor oxidation resistance and relatively low strength of this material have prevented its wide use with new, high specific impulse solid propellants.

Much work is being done in an effort to overcome the poor oxidation resistance of graphite. Most of the more promising approaches involve the use of coatings, additives, or impregnants which diffuse

Walton

Mason

JESSE D. WALTON, JR., is head of the ceramics branch of the Engineering Experiment Station at Georgia Institute of Technology. His work is in the fields of protective coatings for metals, ceramic-metal systems, thermal protective systems and high temperature technology, instrumentation and equipment design.

CECIL R. MASON, JR., assistant research engineer at the Georgia Tech Engineering Experiment Station, directs research on development of high temperature refractory materials, especially fused silica. He has a BS in ceramic engineering from Georgia Institute of Technology.

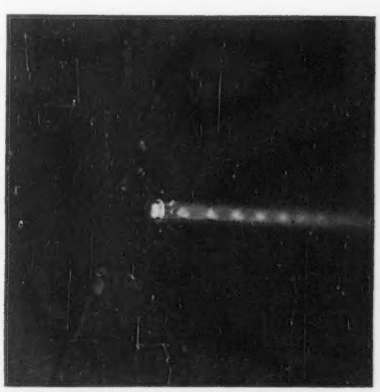

Figure 1—Oxygen-hydrogen rocket motor in operation.

*Submitted for publication January 8, 1960. A paper presented at the 16th Annual Conference, National Association of Corrosion Engineers, Dallas, Texas, March 14-18, 1960.

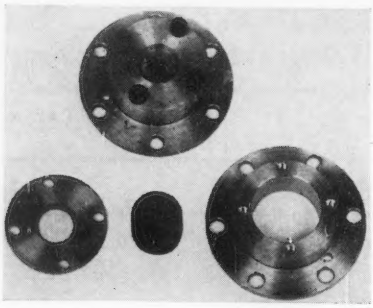

Figure 2—Test nozzle and holder before and after assembly for evaluation in oxygen-hydrogen rocket motor.

Figure 3—Data sheet and pressure-time curve for oxygen-hydrogen rocket motor.

into the graphite to form carbides which are oxidation resistant. One example is silicon coated graphite which, after coating, is heated to form silicon carbide coated graphite. If silicon is added to the graphite prior to forming the desired object, silicon bonded silicon carbide, or silicon carbide bonded graphite may be formed, depending upon whether or not excess silicon is present during the heat treating process.

Approaches of this type are further being expanded into the development of diffusion coatings for the oxidation protection of tungsten and molybdenum.

Experimental Procedure

In order to properly evaluate materials for use in a solid propellant rocket motor exhaust, it is desirable to expose these materials to the actual environments to be encountered. However, due to the high cost of purchasing and firing solid propellant grains it is not economical to use the actual propellant except in the final stages of materials evaluation. Therefore, it is desirable to construct a test facility to simulate solid propellant combustion conditions as closely as possible and at as low a cost as possible. The approach taken at Georgia Tech was that of using a small oxygen-hydrogen rocket motor for preliminary screening followed by the use of a solid propellant motor for final evaluation of promising materials.

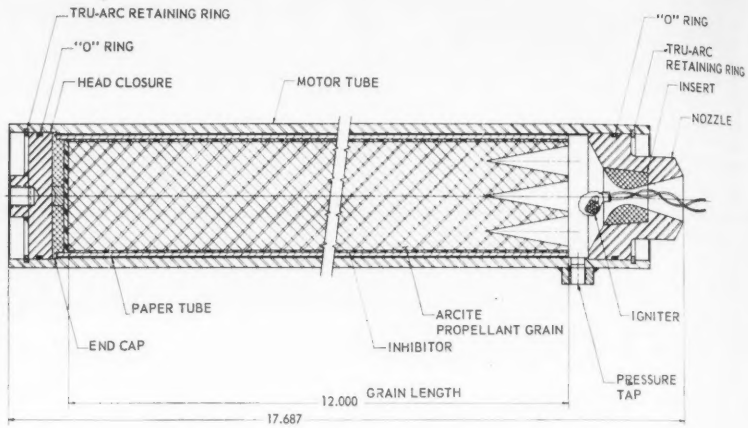

Figure 4—Cross section of solid propellant test motor.

TABLE 1—Operating Conditions of Oxygen-Hydrogen Rocket Motor

Fuel	Gaseous Hydrogen
Oxidizer	Gaseous Oxygen
Fuel-Oxidizer Ratio by Volume	4 : 1
Fuel Flow Rate	36 scf/min
Oxygen Flow Rate	9 scf/min
Upstream Fuel Pressure[1]	860 psi
Upstream Oxygen Pressure[1]	840 psi
Chamber Pressure[2]	275 psi
Starting Chamber Pressure Peak[3]	400 psi
Estimated Flame Temperature	3800 F
Heat Flux in Nozzle Area[4] (Approx.)	1250 Btu/ft²-sec
Exhaust Velocity (Approx.)	7500 fps

[1]Pressure upstream of critical flow orifice.
[2]Continuous chamber pressure using water-cooled nozzle.
[3]Pressure peaks at 400 psi due to external ignition procedure.
[4]Measured through a water-cooled copper nozzle.

Development of Test Facilities

Oxygen-Hydrogen Rocket Motor

In order to provide a facility which could be used for the preliminary screening of solid propellant rocket nozzle materials, a small oxygen-hydrogen rocket motor designed by Battelle Memorial Institute[1] was selected. The operational parameters of this motor are shown in Table 1. Figure 1 shows this motor in operation and Figure 2 shows a test nozzle before and after being cemented into the nozzle holder which is attached to the body of the rocket motor itself.

When the rocket motor is fired with the experimental nozzle a recording is made of the chamber pressure as a function of time. The motor is allowed to continue firing until the chamber pressure drops to 100 psi. Any drop in chamber pressure from that reported in Table 1 (275 psi) is due to erosion and/or corrosion of the nozzle material. The area under the time pressure curve from zero time to the time at which the chamber pressure drops to 100 psi is recorded as the area index (A_{100}) for that particular material. Figure 3 shows a typical time pressure curve for the rocket motor.

From the manner in which the chamber pressure decreases with time some idea may be gained as to the mechanism responsible for the enlargement of the throat area. An immediate drop in chamber pressure upon ignition is usually indicative of either thermal shock or purely mechanical failure, provided of course very refractory materials were used. Were reinforced organic materials used, this could be due to catastrophic decomposition or melting of the material itself. Should the pressure drop off only very slowly over an extended period of time it would be expected that corrosion or volatilization of the nozzle material was responsible.

Solid Propellant Rocket

In the final evaluation of rocket nozzle materials for solid propellant motors it of course becomes necessary to fire these nozzles using such a propellant. Arcite 373 grain was selected for this testing. Although the exact composition and operational parameters of this grain are classified, it can be said that it provides a high temperature, highly erosive and corrosive atmosphere for evaluating rocket nozzles materials.

Figure 4 is a cross sectional drawing of the solid fuel test motor. The insert represents the area in which experimental materials are evaluated. A method similar to that employed with the oxygen-hydrogen rocket motor is used in determining the performance of rocket nozzle materials in this motor. In this case, however, the time pressure curve obtained with the experimental nozzle material is compared on a percentage basis with that obtained when no erosion or corrosion is observed. Thus, the nozzle performance is reported as a percentage of that of a perfect nozzle.

Material Development

Fused Silica

Work on another project involved with the development of materials for nose cones indicated that finely divided fused silica might provide a very promising material for rocket nozzles. The primary properties of fused silica which make it attractive for nose cone use are its low density (1.9 gm/cc), extremely high viscosity at its melting point (3100 F), very low coefficient of thermal expansion which results in phenomenal thermal shock resistance, and its very low thermal conductivity. Since solid propellant rocket nozzle materials must withstand thermal

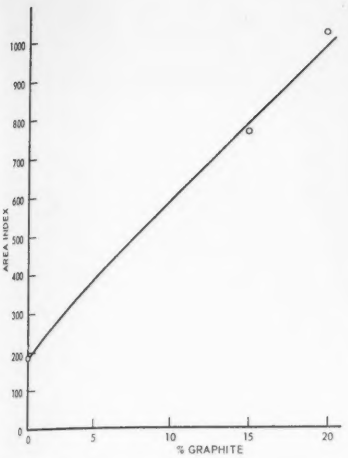

Figure 5—Area index of silica-graphite nozzles as a function of graphite content (to 100 psi).

shock of an extremely severe nature it was thought that fused silica might be used as the basis on which to develop new rocket nozzle materials.

The material used for fabricating objects from fused silica is a mixture of finely divided fused silica suspended in an aqueous dispersion of colloidal silica. Using such a suspension, objects may be formed by slip casting in plaster molds. By mixing coarser particles of fused silica with this suspension a material with the consistency of concrete can be obtained. This mixture may then be used in the fabrication of massive objects. After fabricating, the part is dried and then fired at 2000 F for the period of time required to develop desired strength.

Preliminary nozzles made of fused silica showed that it could withstand the most severe thermal shock environment. However, the relatively low melting point of this material (3100 F) caused it to erode rather rapidly when used on the small oxygen-hydrogen rocket motor. When small additions of finely divided graphite were added to the silica to form a heterogeneous composition, considerable improvement in nozzle performance was noted. Figure 5 plots the area index as a function of graphite content for fused silica nozzles fired on the oxygen-hydrogen rocket motor. Although this represented a means of improving the high temperature resistance of fused silica, the problem was encountered in developing sufficient strength in such heterogeneous compositions. Thus beyond a certain graphite content the graphite itself contributed to weakening the overall composition so that failure was no longer a chemical or thermal one, but rather mechanical in nature. The additions of other refractory materials likewise were limited from a mechanical standpoint. After an intensive program including numerous additives it was determined that fused silica would probably be most suitable as a substructure for some other materials which would be in direct contact with the hot gases.

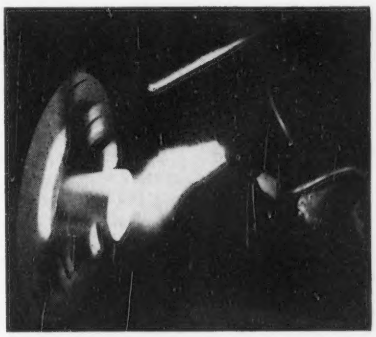

Figure 6—Arc spraying tungsten onto fused silica insert for evaluation in solid propellant test motor.

Composites

Coatings on Fused Silica. One approach which has been taken to develop composites involves the flame spraying or arc plasma jet spraying of very refractory oxides and metals directly onto the slip cast fused silica nozzle inserts. Figure 6 shows the arc spraying of tungsten onto such an insert. Materials which have been successfully arc sprayed directly onto fused silica include zirconia, hafnia, zirconium silicate, hafnium carbide, tungsten carbide, tungsten and tantalum. However the extremely low coefficient of thermal expansion of the silica prohibits the deposition of thick (greater than 15 to 20 mils) coatings onto the inserts. To date no such thin coatings have satisfactorily withstood the conditions generated by the solid propellant rocket motor. Such coatings are virtually impossible to evaluate on the small oxygen-hydrogen motor due to the miniature nature of the nozzles themselves.

Arc Plasma Jet Sprayed Shells. A recent development in the fabrication of thin rocket nozzle insert shells involves arc-spraying the desired material onto a mandrel conforming to the dimensions of the desired insert. After depositing the desired thickness onto this mandrel the mandrel may be removed either by leeching or by mechanically parting the mandrel at the minimum diameter of the nozzle throat. Such a mandrel together with a shell which was arc sprayed onto it are shown in Figure 7.

It is proposed that such shells will be backed up with slip cast fused silica which will give structural support to the shell during the firing of the solid propellant motor.

Ablative Compositions

For certain solid propellant applications where essentially all known materials fail, there is growing interest in the use of ablative materials for nozzles. The primary problem associated with the use of such materials is in obtaining predictable and uniform erosion. These properties are required since the burning characteristics of the propellant would be designed to compensate for erosion as it takes place.

Essentially all available reinforced plastics which might be considered for nozzle applications were evaluated on the

Figure 7—Arc sprayed insert shell and removable mandrel.

oxygen-hydrogen rocket motor. It was found that the slip cast fused silica erodes more slowly and at least as uniformly as the plastic-fiberglass compositions. By modifying the slip cast fused silica with suitable resins it is anticipated that additional erosion resistance may be obtained with increased mechanical strength. A technique has been developed recently which involves the incorporation of a resin in the fused silica suspension which does not interfere with the normal slip casting process. After the desired piece is cast, it is cured at 400 F rather than being fired at 2000 F as in the case of the pure slip cast material. In this condition the resin-bonded fused silica may be machined to close tolerances by conventional machining techniques. It may then be fired to 2000 F to provide a machined, pure ceramic part. However, if the resin-bounded piece is exposed to a rocket motor exhaust, or used as a rocket nozzle it is found to behave very much like the fired object. Thus it is possible that this technique may be employed in the development of materials which would have higher impact strength together with machineability to form either ablative nozzles or back-ups for the refractory shells described previously.

Discussion

Work to date has indicated that the small oxygen-hydrogen motor provides an excellent screening tool for the preliminary evaluation of uncooled rocket nozzle materials. The high temperatures, high gas velocities, and high heat fluxes developed in this motor provide mechanical and thermal shock, and high velocity gas erosion which quickly screen out unsuitable materials. The rocket motor was equipped with a water cooled nozzle in order to provide a high temperature, high velocity gas stream for the evaluation of materials to be used in areas where

aerodynamic heating was contemplated. Materials for guide vanes or jet vanes may likewise be screened with such a facility.

It has been suggested by some investigators using facilities of this type that solid particles might be injected into the motor to simulate the abrasion and to a certain extent the chemical environment found in solid propellant motors. It is probable that additional modification or sophistication of this screening facility may actually defeat the purpose of a test facility of this type. Since the chemistry of the burning of solid propellants and the resulting products exhausted through the nozzle is not entirely understood, synthesized combustion products from a small gaseous motor may not only misrepresent the actual environment to be encountered, but may provide misleading data in the process. It is therefore felt that a gaseous rocket motor such as this is best used as a screening facility to be followed by the actual firing of promising materials in the desired solid propellant motor.

The solid propellant motor incorporating the Arcite 373 grain has been used only a few times in the actual evaluation of experimental compositions. The reason for this is twofold. First, high costs are involved in such tests, and secondly the conditions provided by this motor are so severe that experimental materials have yet to show sufficient erosion and/or corrosion resistance to warrant an extended testing program at this time.

The work on materials for uncooled rocket nozzles has been involved primarily with the use of slip cast fused silica. In the selection of this material, primary consideration was given to thermal shock resistance, high viscosity at its melting point (3100 F), high insulating value, ease of fabrication, and low weight. The fact that pure fused silica eroded fairly rapidly under high temperature, high velocity conditions has suggested that it either be provided with suitable additives to increase its refractoriness and resistance to erosion, or that very refractory shells be placed between the fused silica and the exhaust gases. Of the two approaches, the second appears most promising. When the refractory shell is flame sprayed or arc sprayed directly on the fused silica, difficulty is encountered in developing thick coatings due to the very low thermal expansion of the fused silica.

A more promising approach involves the arc spraying of the desired refractory material of the required thickness onto a removable mandrel. It is then contemplated that this shell will be backed up or supported by the slip cast fused silica. By using this combination of materials very heavy materials such as tungsten could be used only in the thickness required to actually withstand the high temperature environment; this shell would be backed up by the low density fused silica.

It is expected that hafnium oxide or tungsten will be the materials which will offer the most promise of being arc sprayed as a thin shell, and survive the conditions present in the nozzle of the solid propellant motor.

Based on studies using the oxygen-hydrogen motor, it is felt that a suitable back-up material can be developed by combining graphite or some other suitable additive to the slip cast fused silica. The modified slip cast fused silica would provide not only structural support for the tungsten but its high insulating value would protect the motor casing from the high temperatures developed in the nozzle.

As another approach to the development of uncooled rocket nozzle materials it is anticipated that fused silica modified with resins or other suitable additions could be used where ablation of the nozzle can be tolerated, or cannot be eliminated regardless of the material used. The higher specific impulses being developed in solid propellants seems to be invariably accompained by higher temperatures and more erosive and corrosive exhaust gas conditions. Therefore it is not unlikely that ablative type nozzle materials will be seriously considered for use with such propellants. The uniform erosion characteristics together with a reasonably high ablation rate as compared with other ablative materials (fused silica fiber or fiberglass reinforced plastics) make this material particularly attractive. Other advantages which may be derived from using this material are ease of fabrication, low cost, and ability to be formed in large and complicated shapes.

Conclusions

The properties most required of a material to serve as a solid propellant rocket nozzle are: (1) thermal shock resistance, (2) high melting point, (3) corrosion and erosion resistance to the combustion atmosphere, (4) low density and (5) high strength to weight ratio.

In order to evaluate such materials it is necessary to provide as nearly as possible the anticipated rocket exhaust environment. A small oxygen-hydrogen rocket motor has been found most satisfactory for preliminary screening. The more promising compositions are further evaluated using Arcite 373 solid propellant.

Fused silica, finely divided and bonded with colloidal silica, has been evaluated as a rocket nozzle material. The low specific gravity (1.9 gm/cc), excellent thermal shock resistance, high viscosity at its melting point (3100 F), low thermal conductivity, and ease of fabrication of this material led to its selection. However, the relatively high erosion rate of this material, even when modified with refractory additives, suggest the use of this material as a back up for more refractory materials such as tungsten which would be used in the throat section of the nozzle.

The arc plasma jet appears to offer a satisfactory means of forming refractory shells on removable mandrels. Such materials as tungsten, tantalum, hafnium oxide and hafnium carbide have been successfully arc sprayed.

Acknowledgment

Note: This work was carried out under the sponsorship of the Bureau of Ordnance, Department of the Navy, Contracts NOrd—15701 and NOrd—18564.

Reference

1. J. F. Lynch, J. F. Quirk, and W. H. Duckworth. Investigation of Ceramic Materials in a Laboratory Rocket Motor. *Am. Ceram. Soc. Bull.*, 37 (10), 443-445 (1958).

Any discussion of this article not published above will appear in December, 1960 issue.

Effect of Iron, Aluminum and Cadmium Additions On the Performance of Zinc Anodes in Sea Water*

By L. J. WALDRON and M. H. PETERSON

Abstract

Standard hull-mounting zinc anodes of various chemical compositions were immersed in water at the Norfolk Reserve Fleet site and supplied current continuously to a large steel sea wall for 36 weeks. The current was monitored daily during the initial week and 3-4 times weekly thereafter. At the conclusion of the test, the anodes were cleaned, weighed, and analyzed chemically. Electrochemical efficiencies and current outputs were determined.

Data are presented showing the performance of all anodes with graphical representation of typical specimens. Zinc anodes containing iron up to 0.006 percent and small additions of aluminum (to 0.5 percent) and cadmium (to 0.10 percent) outperformed the high-purity zinc anodes (0.0014 percent iron). There was some evidence that the addition of 0.1 percent aluminum without cadmium improves the performance of high-purity zinc. All anodes containing iron up to a maximum of 0.006 percent and aluminum and cadmium in any proportion within the limits of composition tested performed equally well. Minimum contents of 0.025 percent cadmium and 0.1 percent aluminum were indicated. 5.2.2

Introduction

AN EXTENSIVE research and development program by the American Smelting and Refining Company culminated in a recommendation that the composition of zinc anodes for shipboard use be modified to include small percentages of cadmium and aluminum.[1] The results of other tests of zinc anodes have been reported elsewhere.[2,3] The Bureau of Ships of the U. S. Navy Department in cooperation with the American Zinc Institute proposed that the Naval Research Laboratory evaluate the performance of the zinc anodes of different compositions under service conditions. As a result, 24 anodes of 12 compositions were furnished by the American Smelting and Refining Company for tests. The test was started at the Norfolk Group, Atlantic Reserve Fleet test site in May of 1957. The current from each anode was measured periodically until the program was terminated in January of 1958 (36 weeks later.) The total output in ampere-hours, as well as the electrochemical efficiency and chemical analysis of each composition was determined at the end of the test.

Waldron

Peterson

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Test Site

The test site is located at Portsmouth, Virginia, on the Elizabeth River near its merger with the James. The interlocking steel piling used as a retaining wall for the piers was used as a cathode for the test. This sea wall is about 1500 feet long and is made up of individual piles with a height of some 30-40 feet. This height is divided roughly into thirds, one third in the bottom mud, one third in clear water, and one third exposed to the marine atmosphere. Electrical continuity along the 1500 feet of cathode used in the test was assured by welding a one-inch bead at each juncture of the individual pilings. The water resistivity at this site varies seasonally between extremes of 30 and 100 ohm-centimeters. In Table 1 are listed representative resistivity measurements for several dates chosen at random during the test.

Experimental Details

The anodes used in the test were similar in dimensions to the standard 6″ x 12″ x 1¼″ Navy hull zinc and weighed about 22 pounds. Each anode contained two cast-in galvanized steel hull mounting straps.

The anodes were suspended from steel supports along the sea wall and were about 40 feet apart. The anodes were hung from No. 6 copper single conductor cable which served both as a support and as an electrical connection. The anodes were connected to the sea wall through 0.01 ohm shunts which were used for determining the anode currents. The total DC resistances in the metallic paths in

*Submitted for publication October 19, 1959. A paper presented at a meeting of the Northeast Region, National Association of Corrosion Engineers, Boston, Massachusetts, October 6-8, 1958.

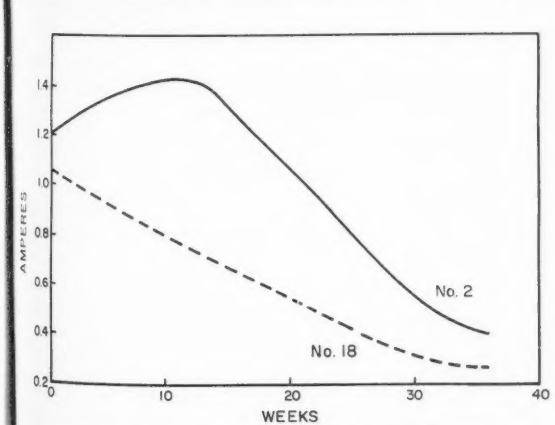

Figure 1—Current output vs time for anodes No. 2 and No. 18.

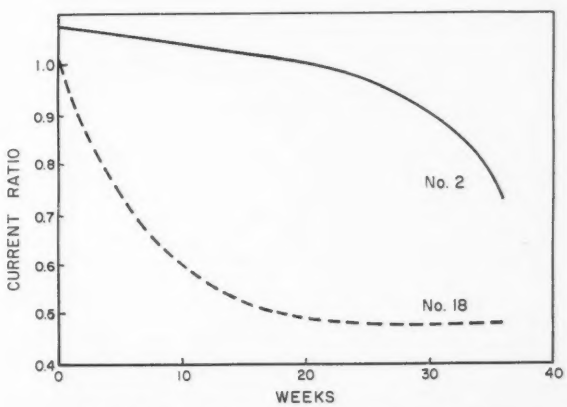

Figure 2—Time effect of ratio of current of zinc to current of magnesium.

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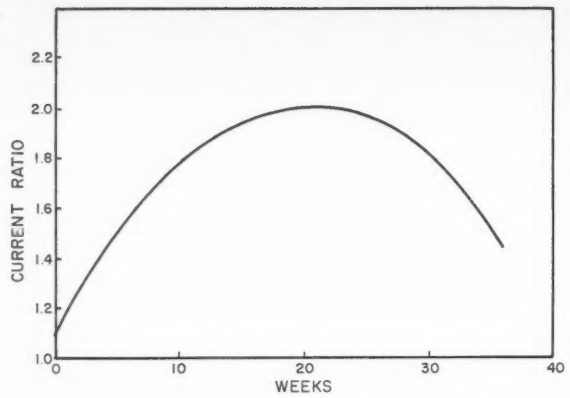

Figure 3—Time effect of ratio of current of anode No. 2 to current of anode No. 18.

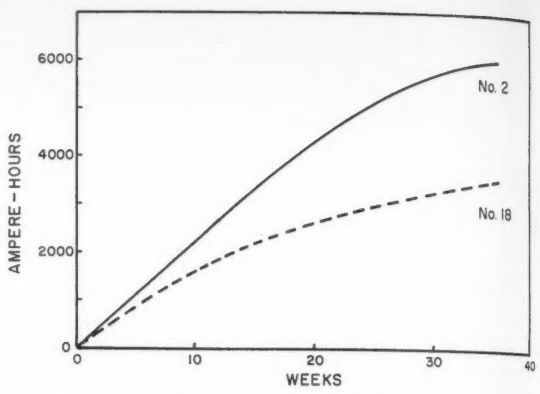

Figure 4—Current output (ampere-hours) vs time for anodes No. 2 and No. 18.

TABLE 1—Resistivity Measurements

Date	Ohm-cm	Elapsed Time, Weeks
27 June 1957	32	5
23 August 1957	32	13
2 October 1957	40	19
21 October 1957	40	23
18 November 1957	44	25
2 December 1957	52	27
9 December 1957	63	28
30 December 1957	67	31
6 January 1958	70	32
13 January 1958	79	33
29 January 1958	86	36

the anode-cathode circuits (including shunt and contact resistances) were measured and found to range from 0.021 to 0.032 ohms.

The current output of each anode was measured daily during the first week of the test, and three to four times per week thereafter. The current from a magnesium anode known to have a practically constant current output that varied only with the water resistivity was monitored as a control. This anode had only one face exposed (50-60 square inches) with all other surfaces coated with plastic. No added resistances were used in the circuit.

Each composition was run in duplicate. All anodes were cleaned and weighed before and after the test; at the conclusion of the test, an anode from each composition was analyzed chemically. These values, together with the nominal compositions as submitted by the manufacturer, are given in Table 2.

To reduce or eliminate the effect that position might have upon the current from the anodes, the anodes were not paired according to composition, but were placed at random positions along the wall. Periodic potential measurements made along the cathode showed no indication of mutual interference or of cathode polarization.

Errors

In practice, it was found that no error was introduced by the assumption that the cathode was a uniform surface. In all cases except one, there was very close agreement between the total ampere-hour outputs of the duplicate samples and no indication that position along the sea wall had any effect. The output in ampere-hours was calculated after each week of test from the average current readings for the period. As no readings were made over the weekend, the average would be heavily weighted toward the middle of the week in the event of significant changes in current during the measuring period (a rare occurrence). In addition no effort was made to compensate for the relatively small variations in current which might have been caused by the tidal action at the site.

Examination of the data, however, indicates that the precision of the experiment was quite good, with excellent agreement between anode pairs. The greatest variation between duplicate anodes over the 36-week test period was about 9 percent while the mean deviation from the pair average was only 1.8 percent. The apparent difference in the current outputs of anodes No. 9 and No. 23 was caused by the cleaning of anode No. 9 midway through the test, thus causing it to show an anomalously high current output. The data from this anode have not been included in any of the calculations.

Experimental Results

The efficiencies of all anodes, determined from comparison of the actual output with the theoretical output, are shown in Table 2. There is some tendency for the two members of a pair to indicate the same efficiency, but as the mean efficiency is 98.9 percent with a standard deviation of only 1.3 percent, it is considered that all anodes had identical efficiencies within the limits of error inherent in the test method, and that the practical efficiency was nearly 100 percent.

The total ampere-hour output produced by each anode is also shown in Table 2. The average total current output of all combinations tested is shown in Table 3. The performance of the low-iron high-purity zincs, Nos. 18 and 24, was poor in comparison with the other compositions tested. This was unexpected.[2,3] However, in this test the two high-purity zincs gave a mean current output of only 3720 ampere-hours compared to a mean current output of 5900 ampere-hours for the fourteen anodes containing varying combinations of aluminum and cadmium.

Zinc containing 0.006 percent Fe + 0.1 Al, and 0.0014 percent Fe + 0.5 percent Al, had a slightly higher total output than the low-iron high-purity zinc but a much lower output than any of the compositions containing aluminum and cadmium. The only non-cadmium composition which performed well was the low-iron zinc containing 0.10 percent aluminum.

Since the performances of all the cadmium-containing alloys were very similar, the following series of curves show only the performance of the two anodes which produced the maximum (No. 2—0.50 percent Al, 0.025 percent Cd, 0.006 percent Fe) and minimum (No. 18—no Al, 0.001 percent Cd, 0.0014 percent Fe) output of current.

Figure 1 shows the current produced by each of these anodes as a function of time. While there is a marked difference in the instantaneous current for any given day, this difference is somewhat obscured because the current from both anodes decreased after approximately 13 weeks due to seasonal changes in the water resistivity.

To show the decrease in current due to filming of the low-iron high-purity anode (No. 18) more clearly, the ratio of the currents produced by the zinc anodes to that of the magnesium anode was plotted. Experience with the magnesium anode had shown that the only variable which affected its current output was water resistivity. The resulting curve, Figure 2, is at least approximately independent of the water resistivity and shows the reduced current from anode No. 18 due to filming of its surface. The reduced current from anode No. 2 after 25 weeks could be due to filming, a reduction in anode area, or both. The behavior of these two zincs is also shown by another comparison in Figure 3. Here the ratio of the current produced by the two anodes is plotted as a function of time. This curve shows the same general trends as are shown in Figure 2.

In Figure 4 the cumulative ampere-hour outputs of anodes No. 2 and No. 18

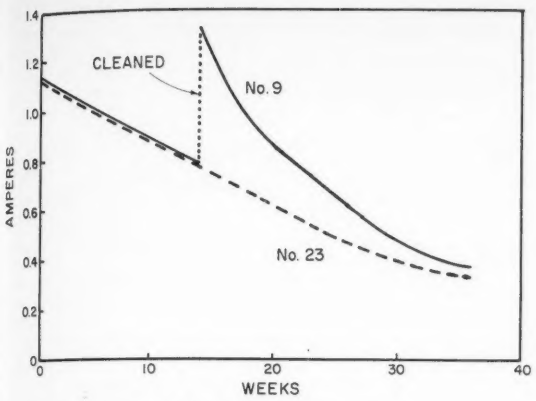

Figure 5—Effect of cleaning on current output.

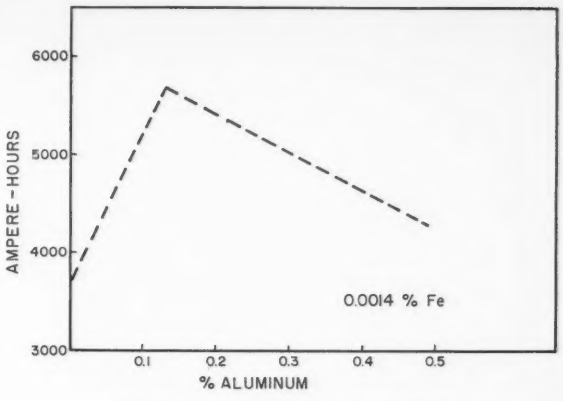

Figure 6—Effect of aluminum on current output.

TABLE 2—Analysis, Current Outputs, and Electrochemical Efficiencies of all Anodes Tested

Sample No.	Mfgrs. No.	Nominal Composition—Percent			Chemical Analysis—Percent			Total Output, Ampere Hours		Efficiency, Percent
		Al	Fe	Cd	Al	Fe	Cd	Theoretical	Measured	
24	6810	None	0.0014	Low	....	0.0010	.....	3967	3895	98.2
18	6840	None	0.0014	Low	0.005	0.0014	0.0001	3560	3546	99.6
25	527	0.10	0.0014	Low	0.127	0.0022	0.0007	5958	5868	98.5
10	528	0.10	0.0014	Low	....	0.0024	.....	5511	5489	99.6
13	6860	0.50	0.0014	Low	....	0.0011	.....	4275	4204	98.3
20	6870	0.50	0.0014	Low	0.49	0.0016	0.0008	4490	4318	96.2
22	578	0.10	0.005	Low	....	.....	.....	4617	4492	97.3
7	579	0.10	0.005	Low	0.101	0.006	0.0003	4545	4478	98.5
23	653	0.50	0.005	Low	0.50	0.006	0.0004	4253	4089	96.1
9	654	0.50	0.005	Low	....	0.006	.....	4952*	4855	98.0
1	583	0.10	0.005	0.025	0.097	0.007	0.021	5661	5667	100.1
17	584	0.10	0.005	0.025	....	.....	.....	5949	5930	99.7
11	588	0.10	0.005	0.05	0.095	0.006	0.049	5777	5806	100.5
5	589	0.10	0.005	0.05	....	.....	.....	6057	6037	99.7
21	593	0.10	0.005	0.10	0.103	0.007	0.105	5876	5820	99.0
8	594	0.10	0.005	0.10	....	.....	.....	5850	5803	99.2
12	658	0.50	0.005	0.025	0.51	0.006	0.025	5783	5813	100.5
2	659	0.50	0.005	0.025	....	.....	.....	6070	6040	99.6
3	663	0.50	0.005	0.05	0.50	0.006	0.057	6012	5942	98.8
4	664	0.50	0.005	0.05	....	.....	.....	5965	5931	99.4
19	668	0.50	0.005	0.10	0.49	0.006	0.10	6042	5920	98.0
14	669	0.50	0.005	0.10	....	.....	.....	6196	6019	97.1
6	537	0.10	.....	0.05	....	.....	.....	5763	5813	100.9
16	538	0.10	.....	0.05	0.13	0.0015	0.06	5943	5963	100.3

* Cleaned.

TABLE 3—Average Ampere-Hour Output Obtained for all Anodes

Cadmium, Percent	Average Ampere—Hour Output For Anodes			Iron—%
	0.5% Al	0.1% Al	No. Al	
0.001	4261	5678	3720	0.0014
0.001	4089	4485	....	0.006
0.025	5926	5798	....	0.006
0.05	.....	5888	....	0.0014
0.05	5936	5921	....	0.006
0.10	5970	5811	....	0.006

have been plotted as a function of time. Here again it is quite evident that the aluminum- and cadmium-containing anodes have far outperformed the high-purity zinc anode.

The effect on the current output of cleaning the surface of one anode (No. 9—0.5 percent Al, 0.005 percent Fe, low Cd) is shown in Figure 5. Removing the film from anode No. 9 after fourteen weeks' exposure increased its current output by approximately 80 percent. However, with continued exposure, its current dropped rapidly, approaching that of anode No. 23 at the end of the test.

The effects of aluminum additions to low-iron high-purity zinc are summarized graphically in Figure 6. A maximum ampere-hour output was obtained at a concentration of aluminum somewhere between 0 and 0.5 percent. An insufficient number of samples were run to determine definitely the exact value of this maximum.

Figure 7 shows the effect on ampere-hour output of cadmium additions to high-iron (0.005 percent) zinc anodes containing 0.1 percent and 0.5 percent aluminum. Here the addition of 0.02 percent cadmium to either series of alloys greatly improved their performance. Increasing the cadmium content to 0.10 percent did not further improve the performance.

Figures 8 and 9 show the appearance of anodes No. 6 and No. 19 at the end of the test before their surfaces were cleaned. The surface of these and of most of the anodes which gave maximum performance was relatively free of adher-

ent corrosion products. Any films remaining on the anodes at the time they were removed from test were removed easily by shaking or gently washing. Note the uniform corrosion of the surface of these anodes. This was characteristic of all the anodes which gave high current outputs.

Figure 10 shows the appearance of anode No. 23 as removed and before cleaning. Its surface is completely covered with dense adherent corrosion products. This condition was typical of all anodes which gave low current outputs. Figure 11 shows the appearance of anode No. 24 after cleaning. The surface of the metal was nonuniformly corroded and the corrosion products were tightly adherent and were removed with difficulty.

Summary and Conclusions

A test of twelve pairs of zinc anodes of several compositions has demonstrated that anodes containing small additions of aluminum and cadmium gave higher total current outputs than high-purity zinc anodes. There was some evidence that the addition of 0.1 percent aluminum will improve the performance of high-purity zinc (0.0014 percent Fe maximum).

The current outputs of all anodes decreased with time. This was obscured by the fact that the water resistivity at the test exposure site was increasing due to seasonal effects. However, a comparison of the effect of time on current output of several zinc anodes, both direct and with a magnesium anode used as a control, indicated that the decreased current of those anodes which gave poor performance was due to filming of the anode surface with adherent corrosion products. It is believed that the reduced current output of those anodes which gave satisfactory performance was due largely to changes in anode dimensions.

Within the limits of error of this test, all anodes containing iron up to a maximum of 0.006 percent, and cadmium and aluminum in any proportion, performed equally well, indicating that the aluminum and cadmium are beneficial and that their content is not critical within the limits of compositions tested. Minimum contents of 0.025 percent Cd and 0.1 percent Al were indicated.

Within the limits of error in determining the efficiency of the anodes, all anodes tested showed a practical electrochemical efficiency of 100 percent.

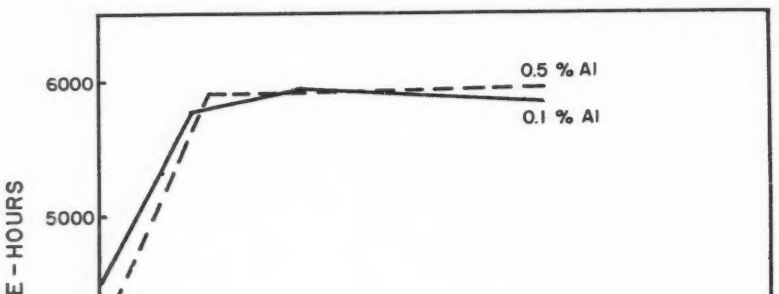

Figure 7—Effect of cadmium on current output.

Acknowledgment

The cooperation of the officers and men of the Norfolk Group, Atlantic Reserve Fleet, in this work is gratefully acknowledged.

References

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2. R. B. Teel and D. B. Anderson. The Effect of Iron in Galvanic Zinc Anodes in Sea Water. *Corrosion*, 12, 342t (1956) July.
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DISCUSSION

Comments by J. A. H. Carson, Pacific Naval Laboratory, Esquimalt, British Columbia:

The authors suggest in their paper that the reduction in current output with time for the better anodes, for example anode

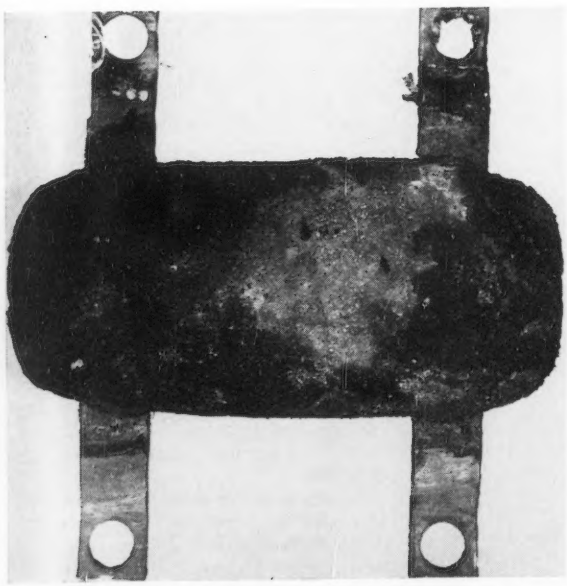


Figure 8—Anode No. 6 as removed from test.

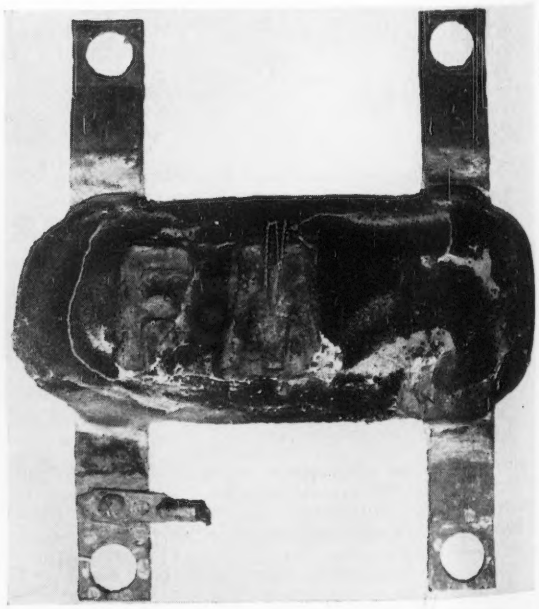


Figure 9—Anode No. 19 as removed from test.

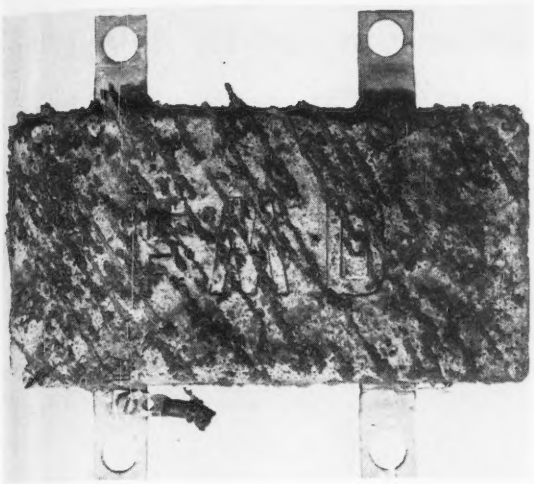


Figure 10—Anode No. 23 as removed from test.

Figure 11—Anode No. 24 after cleaning

No. 2, could be attributed to a combination of the effects of the increase in water resistivity and the decrease in size of the anode. It is most important to be able to distinguish between a loss of output because of this increase in electrolyte resistance and a loss due to other causes such as anode polarization by corrosion products. The following calculations based on an equation for electrolyte resistance developed by Dwight,[1] indicate that the decreased output of anode No. 2 was primarily due to the change in electrolyte resistance.

Dwight's Equation:

$$R = \frac{\rho}{2\pi L}\left(2.303\left(\frac{\log 2L}{r}\right) - 1\right)$$

where R = electrolyte resistance
ρ = electrolyte resistivity in ohms-ft.
L = length of cylindrical anode in ft.
r = radius of cylindrical anode in ft.

In order to use Dwight's equation the rectangular section of the anode must be converted to its equivalent circular section. From some analog work carried out at our laboratory the factors by which the perimeter of the rectangular sections must be multiplied to get the circumferences of the equivalent circular anode sections are: 0.8 for a 1 in. × 5 in. section at 13 weeks, and appromixately .75 for a ½ in. × 4 in. anode section at 36 weeks.

At the 13 week point the parameters in the above equation for an anode 1 in. × 5 in. × 11 in. would therefore be: $\rho = 1$ ohm-ft., $L = \frac{11}{12}$ ft., and $r = \frac{2(1+5)}{2\pi(12)}(0.8) = \frac{1.5}{12}$ ft.

From which R = .29 ohms.

For the approximately ½ in. × 4 in. × 10 in. anode at the 36 week point, the parameters would be: $\rho = 2.7$ ohm-ft., $L = \frac{10}{12}$ ft., and $r = \frac{2(4 \div \frac{1}{2})}{2\pi(12)}(0.75) = \frac{1.1}{12}$ ft.

From which R = .98 ohms.

Adding .02 ohms lead resistance, the total circuit resistance for the No. 2 anode equals 0.31 ohms at 13 weeks, and 1.0 ohms at 36 weeks.

If the cathode is unpolarized and at approximately —0.6 volt vs. a silver-silver chloride reference electrode, then the net driving potential would be 0.45 volt and the anode current output would be:

$$\text{At 13 weeks } \frac{.45}{.31} = 1.45 \text{ amps.}$$

$$\text{and at 36 weeks } \frac{.45}{1.0} = 0.45 \text{ amps.}$$

These figures coincide quite closely with the actual measured values and indicate that the decreased output can be attributed to the increase in electrolyte resistance alone. This conclusion is in accordance with some work[2] in which this writer participated, which indicated that in 2 months there was an insignificant polarization effect from the corrosion product films on zinc alloys of comparable composition to those described in this paper.

References

1. H. B. Dwight. Calculation of Resistances to Ground. *Electrical Engineering* 1319 (1936) Dec.
2. J. A. H. Carson, W. L. M. Phillips and J. R. Wellington. A Laboratory Evaluation of Zinc Anodes in Sea Water. *Corrosion*, 16, 171t-177t (1960) Apr.

NOTE: The opinions expressed in this paper are those of the authors and not necessarily those of the Naval Research Laboratory or of the Navy Department.

Any discussion of this article not published above will appear in December, 1960 issue.

Oxidation and Corrosion of Zirconium and Its Alloys*

By B. COX

Abstract

In contrast to the mode of breakdown in Zircaloy-2 and carbon-melted zirconium the oxide film on arc-melted zirconium corroded in steam or water at 300 C fails initially as a result of more rapid oxidation along grain boundaries. Stresses set up in the oxide film by this preferential attack along grain boundaries cause cracking of the film parallel to the grain boundaries. Subsequent failure within the grains causes the final disintegration of the oriented protective oxide and its replacement by a loose coating of unoriented ZrO_2. 6.3.20

Introduction

IN AN earlier paper[1] a comparative study was presented of the effect of discrete carbide inclusions in carbon-melted zirconium and Zircaloy-2 on the breakdown of the protective oxide film during corrosion in steam and aqueous solutions at 300 C. A similar study of oxide film breakdown in arc-melted zirconium and Zircaloy-2 has been carried out. The part of the latter study related to arc-melted zirconium is presented here. Most of the data available on the corrosion of zirconium in steam and water have been obtained from studies of the behavior of arc-melted crystal-bar zirconium.[2] There is little data on the kinetics of corrosion of arc-melted sponge zirconium owing to its sensitivity to the presence of small quantities of impurities.

The kinetics of the corrosion reaction in zirconium alloys fall into two parts. Initially there is a period of protective film formation which follows a law of the form $\Delta m = kt^n$ where Δm is the weight gain, t the time, and k and n are constants. The constant n has a value less than 0.5 during the initial period of protective film formation showing that the process is here diffusion controlled. After a time the corrosion rate increases and the metal becomes covered with a white non-adherent oxide; the exponent n during this period is approximately 1 indicating a linear corrosion rate.

★Submitted for publication October 21, 1959.

About the Author

BRIAN COX is now conducting studies on the corrosion of zirconium and other metals at the Atomic Energy Research Establishment, Harwell, Berks., England. He joined the UKAEA in 1955 after receiving a PhD in chemistry from Cambridge University, having previously obtained his BA at the same university. He has previously published papers on the structures of inorganic complex fluorides.

The onset of "breakaway" or "transition," as this change in rate law is called, has yet to be satisfactorily explained. It has been said to occur at a critical oxide thickness, which is a function of temperature. At 300 C this critical film thickness is approximately 30 mgm/dm² for Zircaloy-2. Cracking of the oxide film at this point due either to stresses built up by matching of the oxide and metal lattices or to a phase transformation in the oxide have variously been proposed as the mechanism by which this failure takes place.

Schwartz, Vaughan and Cocks[3] in a study of crystal bar zirconium noted that the breakaway appeared to spread from inclusions which they identified as zirconium hydride; they also found that whereas some parts of the surface were showing white oxide, others still showed interference color films. In an earlier paper[1] it was seen that the concept of the critical film thickness for breakaway is meaningless in the case of alloys containing precipitated phases in the metal surface, the factor determining the time of "breakaway" of such material being dependent on the rate of propagation of oxide film failure from nuclei which are formed at a very early stage in the corrosion.

The question of nucleation and propagation of oxide film breakdown in material containing no precipitated phases has not been examined previously. It was expected that a study of the corrosion of arc-melted sponge zirconium would provide useful information for comparison with the previous study of carbide inclusions in zirconium and Zircaloy-2[1] and a

TABLE 1—Analysis of Arc-melted Zirconium Billet Z/208

Element	Content
Hf	0.07 Percent
Fe	325 ppm
Ni	16 ppm
Cr	55 ppm
N_2	100 ppm
O_2	0.1 Percent
H_2	20 ppm
Sn	12 ppm
Al	300 ppm
Mg	10 ppm
Pb	100 ppm
Mn	5 ppm
Cu	10 ppm
Sb	30 ppm
Ge	10 ppm
K	50 ppm
Zn	<100 ppm
Rb	< 20 ppm
As	< 10 ppm
Ti	< 10 ppm
Bi	< 10 ppm
Co	< 10 ppm
In	< 10 ppm
Mo	< 10 ppm
Ag	< 10 ppm
Sr	< 10 ppm
V	< 10 ppm
Be	< 2 ppm
Ga	< 2 ppm
Au	< 1 ppm
Cd	< 0.2 ppm
Li	< 0.1 ppm

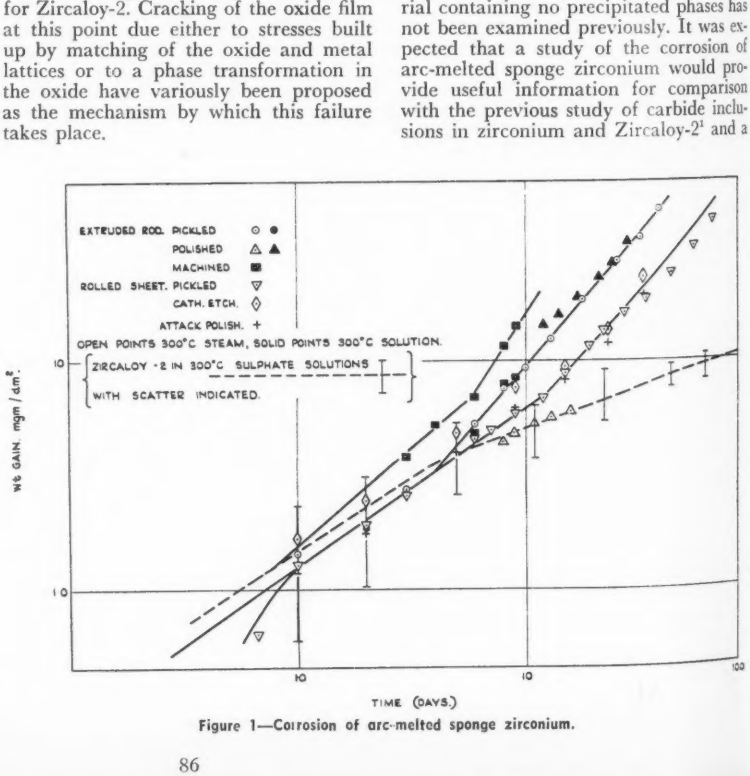


Figure 1—Corrosion of arc-melted sponge zirconium.

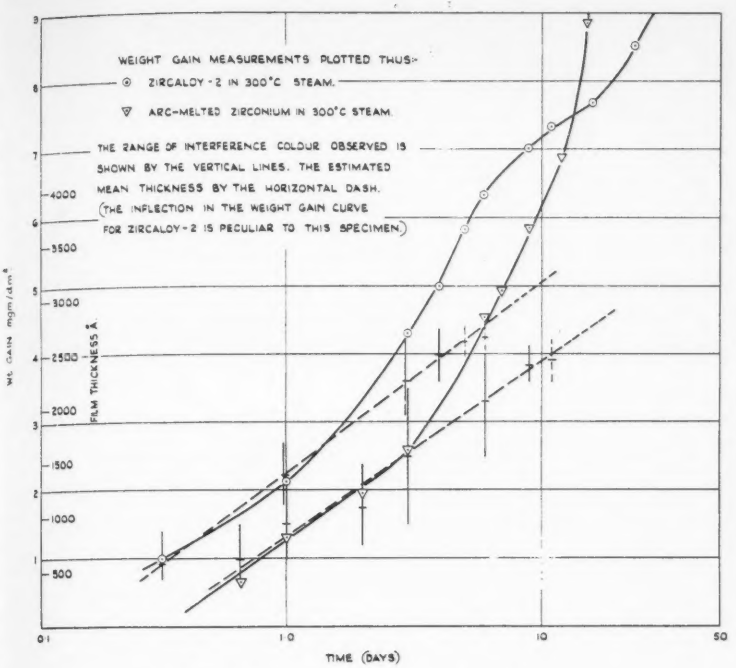
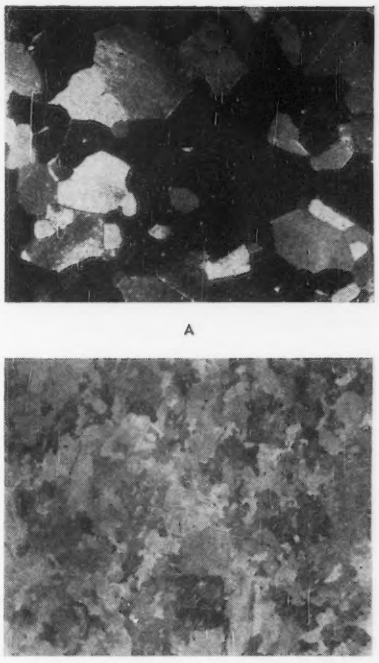

WEIGHT GAIN MEASUREMENTS PLOTTED THUS:-
⊙ ZIRCALOY-2 IN 300°C STEAM.
▽ ARC-MELTED ZIRCONIUM IN 300°C STEAM.
THE RANGE OF INTERFERENCE COLOUR OBSERVED IS SHOWN BY THE VERTICAL LINES. THE ESTIMATED MEAN THICKNESS BY THE HORIZONTAL DASH.
(THE INFLECTION IN THE WEIGHT GAIN CURVE FOR ZIRCALOY-2 IS PECULIAR TO THIS SPECIMEN.)
Wt. Gain mgm/dm²
FILM THICKNESS Å
TIME (DAYS)
0·1
1·0
10
50

Figure 2—Film growth estimated from interference colors.



A

B

Figure 3—Optical micrographs of arc-melted sponge zirconium (billet Z208) in rolled (annealed) and extruded condition. A—Rolled sheet, polarized light, approximately 260X; B—extruded rod, polarized light, approximately 145X.

parallel study of arc-melted Zircaloy-2 and crystal-bar zirconium.

Experimental

The material chosen for this study was from an arc-melted sponge zirconium billet Z208. An analysis of the material is given in Table 1. This material was available both as extruded rod and as annealed rolled sheet; specimens from both batches were studied. Previous work[4] indicated that this material had good oxidation resistance in air compared with other examples of arc-melted zirconium, and data on its corrosion resistance in 300 C steam at atmospheric pressure and 300 C sulfate solutions (0.08 m K_2SO_4; 0.02 m H_2SO_4; 0.005 m $CuSO_4$) was obtained (Figure 1).

Specimens were prepared by the following methods:

1. Machined Finish.
2. Machined, abraded to 3/0 emery and pickled in HF/HNO_3.
3. Machined, abraded to 3/0 emery and polished on alumina laps.
4. Machined, abraded to 3/0 emery and attack polished.[5]
5. Machined, abraded to 3/0 emery attack polished and cathodically etched.[6]

Replicas were prepared from specimens at suitable time intervals using a two-stage formvar-carbon process; these were shadowed with gold-palladium alloy at approximately 30 degrees. Replicas were examined using a Philips EM 100 electron microscope. Replication of specimens used to obtain weight gain data was avoided where possible after the onset of breakaway owing to the extraction of measurable quantities of loose oxide in the replicating process. Titanium high pressure equipment has been used at all times to obtain the maximum possible cleanliness of specimen surfaces. The equipment and experimental methods used in obtaining the weight change data for aqueous solutions will be described elsewhere.[7]

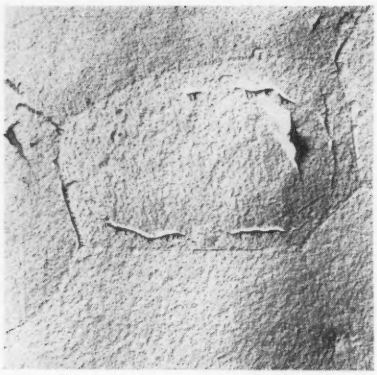
Results

The weight change results showed good correlation between the steam and solution corrosion rates at 300 C (Figure 1). There is no significant difference between the results for corrosion in aqueous solution and steam, and no large variations due to the method of surface preparation have been found. The small differences present are within experimental error when the spread obtained on results for arc-melted Zircaloy-2 in solution is taken into account (Figure 1). Similarly the apparently shorter time to breakaway of the extruded rod (not annealed) compared with the rolled sheet (annealed) is not significant when the variation in time to breakaway for similar specimens is taken into account.

Visual observation at low magnification showed that the rate of film growth varied appreciably depending upon the orientation of the grains, differences in film thickness up to a factor of 2 being observed. The mean oxide thickness was estimated from the interference colors shown by the individual grains and followed closely estimations of the corrosion rate of Zircaloy-2 from observations of the interference colors shown by the oxide (Figure 2). Figure 3 shows samples in both the rolled and the extruded condition.

The rate of film growth estimated from the interference colors decreased rapidly and a limiting thickness of about 2500-3000 Å was reached. Subsequently the interference colors became less distinct and it was not possible to decide whether the thickness was constant or changing slowly. Interference colors were estimated by comparison with a color chart prepared by anodizing a strip of zirconium in ammonium borate solution. The mean film thickness of the surface was estimated microscopically from a rough determination of the numbers of grains in different interference color ranges. Visual comparison of specimens with the anodization chart at low magnification is unsatisfactory as the eye sees the brightest colored grains preferentially and does not see a mean interference color. The limiting interference color thickness was equivalent to that produced by anodization at about 120-125V and on subsequent corrosion the film became dark and did not continue to follow the interference sequence obtained by anodization. Microscopic observation showed that in the case of arc-melted sponge zirconium this darkening commenced at the grain boundaries and remained for some time as a band of dark oxide following the grain boundaries. Some time after they were first observed these bands began to show lines of grey following the grain boundaries under dark field illumination and these became more prominent with increasing time.

The appearance of the band of thicker oxide along the grain boundaries corresponded very closely in time with the change in corrosion kinetics, although the grey/white oxide along the grain boundaries was not visible until several days later (first noticed after 19 days on the rolled sheet). Using the electron



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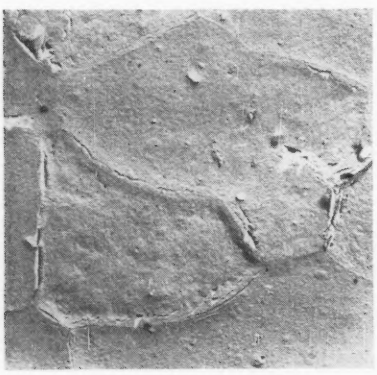
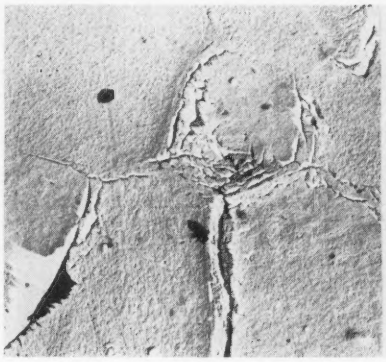


B

Figure 4—Electron micrographs of rolled zirconium sheet showing the initial stage of cracking parallel to grain boundaries. A—High magnification showing relation of cracks to grain boundaries, approximately 5500X; B—Low magnification showing formation of ridges of thicker oxide along boundaries, approximately 2100X.



A—Approximately 2700X

B—Approximately 5400X

Figure 5—Electron micrographs of rolled zirconium sheet showing the development of the cracking along the grain boundaries and onset of general cracking in some grains.

A—Approximately 2000X

B—Approximately 2000X

Figure 6—Electron micrographs of extruded arc-melted zirconium showing an advanced stage in the cracking of the oxide film. At the low magnification the ridges along the grain boundaries are clearly visible.

microscope, however, it was found that cracking of the oxide parallel to the grain boundaries had commenced at a point prior to the change in kinetics; the ridges of thicker oxide along which the cracks ran were also clearly visible at relatively low magnification (2000-3000X). This cracking was very slight at this stage (Figure 4) and it appeared that, within experimental error, the onset of more severe cracking (Figures 5 and 6) marks the "transition" or "breakaway" point as detected by weight change measurement in arc-melted zirconium. At this point cracking is sufficient to permit measurable growth of oxide under the protective film, more severe cracking rapidly ensues as a result of the stresses produced and the white oxide subsequently appears growing through the cracks.

At a later stage more general failure of the oxide takes place within the grains (Figures 6 and 7) and the surface becomes progressively grey and then white to the naked eye. That the formation of the thicker oxide ridges leads to the cracking of the film rather than vice-versa is shown in Figure 8 where ridges are clearly visible at the grain boundaries prior to the formation of cracks. The commencement of cracking along some of the ridges can be seen in Figure 8A. The breakdown of the oxide film on the extruded rod follows an identical pattern but observation of it with the optical microscope is rendered more difficult by the microstructure of this material (Figure 3); using the electron microscope it is found that the course of the breakdown is identical with that of the rolled sheet. Non-uniform attack over the grain surface was not prominent prior to the darkening of the oxide film, and a search of subsequent specimens produced only a few grains showing bands of higher order interference colors at grain boundaries prior to the onset of darkening; this number was very much less than the number subsequently found to show thicker oxide at the grain boundaries on electron microscope examination.

The preferential oxidation along chosen grain boundaries is thought to be due to enhanced diffusion through the oxide at these points due to mis-match of the metal lattices at the boundaries involved. Thus the severity of cracking and its time of onset might be determined by the relative difference in orientation of the different grains. Other factors influencing the stresses along any boundary would be the rate of the film growth on the grains involved and the presence of any step at the grain boundary initially (e.g., steps produced by cathodic etching of specimens).

An attempt was made to estimate which of these factors predominated and hence to predict the boundaries along which failure would first occur. The relative orientations of groups of grains were assessed from their extinction angles under polarized light and the rate of film growth on the individual grains was estimated from the interference colors shown after 2 days oxidation.

Two specimens were studied, one attack polished and the other cathodically etched, and in the latter instance the relative step height at grain boundaries was estimated from the prominence of the grain boundary on a bright field optical micrograph. Electron microscopic examination of these specimens showed that cracking commenced after 4-5 days in steam at 300 C (Figure 4). In the case of the cathodically etched specimen the determination of the causes of the initial cracking were confused as a result of the steps produced in the cathodic etching. Cracks almost invariably formed along boundaries having large steps irrespective of the oxide film thickness on either side of the boundary; observations of adjacent grains not showing steps at their boundaries, however, indicated that misorientation at the grain boundary was more important than rate of film growth on the grains involved. Examination of the attack polished specimen, which showed no steps at grain boundaries ini-

A

B

Figure 7—Electron micrographs showing the difference in appearance of two specimens at approximately the same point in the film breakdown process. A—Pickled specimen on which the grain structure was clearly visible initially, approximately 5000X; B—Polished specimen on which a uniform Beilby layer was present initially, approximately 2500X.

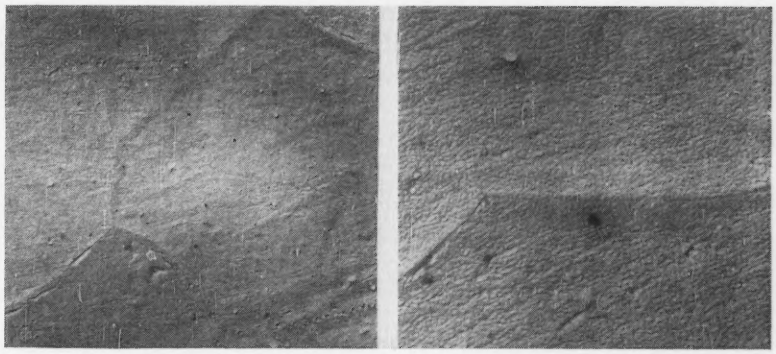


Figure 8—Electron micrographs of rolled sheet showing the presence of ridges along grain boundaries prior to cracking of the oxide film. Left specimen is at a magnification of approximately 2100X; right specimen at 7000X.

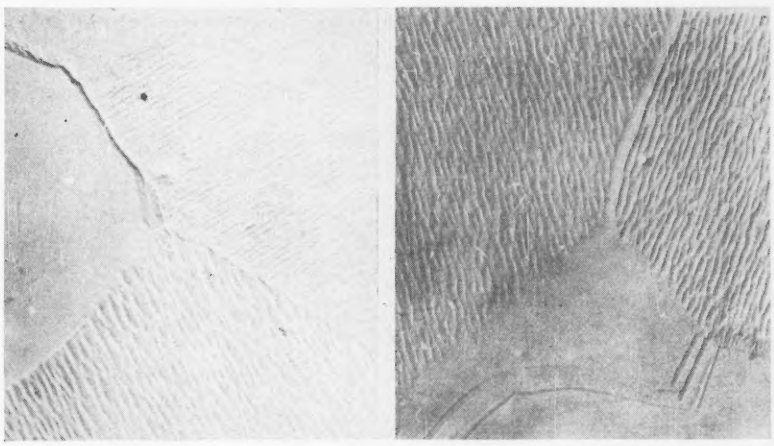


Figure 9—Electron micrographs of rolled zirconium sheet after cathodic vacuum etching showing the uniformity of structure developed throughout the grains, the steps produced at grain boundaries and the absence of any precipitated impurities in the grain boundaries. Both illustrations are at a magnification of 5600X.

tially, confirmed this. The initial cracking was due largely to formation of ridges along boundaries showing large misorientations although some cracking was observed along boundaries where the difference in oxide growth rate was sufficient to produce a noticeable step at the boundary. Subsequent failure within grains occurred first on those grains showing the thickest oxide films. Thus there were some grains which showed uniform darkening of the oxide at the time when only grain boundaries were showing dark bands. These grains were the first to show internal failure following the general grain boundary failure.

The examination of the cathodically etched specimen indicated that the factors considered above are not independent; thus the boundaries showing the largest steps probably also show large differences in orientation and may show large variations in film growth rate. In these cases it is impossible to separate the factors producing cracking. From the observation of grains showing little difference in film growth rate and no steps at grain boundaries, however, it appears that enhanced diffusion along the grain boundaries due to differences in orientation of the grains is a significant factor. Segregation of impurities in the grain boundaries, if it were the cause of enhanced oxidation, would be expected to operate equally along all boundaries. This is not found in practice and electron micrographs of cathodically etched specimens fail to show any visible evidence of segregation (Figure 9).

Discussion

The breakdown of the oxide film on arc-melted zirconium appears to be explicable solely on the basis of the mechanical failure of the oxide film as a result of stresses produced during its growth. The initial failure takes place along grain boundaries where the stress is raised as a result of enhanced local film growth. This enhanced film growth along the grain boundaries is due to more rapid diffusion through the oxide at the grain boundaries probably as a result of mismatch of the lattices of adjacent grains rather than as a result of segregation of impurities in the grain boundaries. Large differences in the oxidation rates of adjacent grains are apparently responsible for cracking in some other instances. Diffusion along grain boundaries has been found to be several orders of magnitude greater than in the bulk of the material;[8] however phenomena resulting from this seldom occur more than 2-3 times as rapidly at the grain boundary as in the bulk of the grain owing to lateral diffusion away from the boundary itself which is very thin. Since the thin oxide film on zirconium alloys is oriented relative to the metal surface[9] it would be expected that mismatch of the oxide lattice would also occur at grain boundaries and that diffusion along these boundaries would be greater than through the bulk of the oxide film. In like manner although this diffusion might be very much enhanced the additional film growth produced would probably not be greater than a factor of 2-3 due to lateral diffusion of oxygen and the narrow width of the diffusion path.

It seems surprising in view of this that enhanced film thickness at the grain boundaries was not found extensively in the interference color range but only became obvious when the rate of growth of interference color film had decreased almost to zero and the darkening of the oxide commenced. Several aspects of the behavior of zirconium and its alloys, especially Zircaloy-2, appear to center on this point at which the thin interference color film changes to the dark protective film which persists until breakaway in the case of Zircaloy-2. Thus the kinetics of film growth in the interference color region appear to differ from those in the black film region for all the

materials considered. The experimental data do not give a straight line log W/log t plot and these together with observations of the change in interference color suggest that the initial film formation may obey a logarithmic law rather than a parabolic one (Figure 2).

After growing to about 2500-3000 Å (4.1-4.8 mgm/dm²) further increase in the film thickness is difficult to estimate from interference colors, especially since the film becomes darker and the interference colors progressively fade out. The exponent of the log/log plot decreases to about 0.3 in this region in the case of Zircaloy-2. This decrease in the exponent in the rate law does not occur with arc-melted zirconium and the cracking of the oxide film with the resultant rapid corrosion probably masks any effect which takes place. The difference in the behavior of arc-melted zirconium and Zircaloy-2 derives initially from this change in the nature of the oxide film at the end of the interference color region. Thus in the case of zirconium, darkening (i.e., thickening) commences at the grain boundaries thereby producing stresses which lead to the failure of the film, whereas in Zircaloy-2 the darkening is uniform over the whole surface within the resolution of the optical microscope.

The nature of this darkening process is not clear but from the fact that the interference colors remain faintly visible for some time afterwards and that in the case of arc-melted zirconium it produced ridges in the oxide leading to failure, it may be infered that the darkening originates below the interference color film at the oxide-metal interface. In the case of Zircaloy-2 and other alloys this dark film[9] has been shown to be oriented monoclinic ZrO_2. The nature of the interference color film formed initially, whether by oxidation in water, steam or oxygen, is in some doubt and reports that it is cubic (or tetragonal) have appeared[2, 10, 11] although in other instances it is stated to be monoclinic.[9] Observation of the structures of these thin films *in situ* is restricted to glancing angle electron diffraction and the discrepanices between the various reports may be the result of orientation effects in the oxide as the three lattices involved are very similar.[2] Electron diffraction studies of thin films on the arc-melted zirconium used in this work have indicated the presence of cubic or tetragonal ZrO_2, but taking into account the extra unidentified rings which are always present and the fact that the 111 line is generally broader than would be expected although not resolved into 111 and $11\bar{1}$ it would be unwise to assume that similar diffraction patterns could not be produced by oriented monoclinic ZrO_2; X-ray studies of thicker films have confirmed the presence of monoclinic ZrO_2 oriented with respect to the metal surface.

Despite the doubts about the structure of the initial interference color film this would seem to be less important than the change in appearance from a transparent oxide film to a dark (almost black) oxide. In materials which are normally white in bulk this black appearance is usually associated with considerable non-stoichiometry. Diffusion through the initial film has been shown to be due to oxygen diffusion via anion vacancies[2] and the change in color of the film may be associated with a progressive increase in the non-stoichiometry of the oxide as a result of the vacancy gradient present in the thickening film. This non-stoichiometry would be greatest at the oxide-metal interface and this might give rise to the observation that the dark film appears to grow under the interference color film. The interference colors would still be visible in the outer film for a short while but the increase in non-stoichiometry would progressively destroy the internal reflection on which the interference phenomenon depends, leading to the observed fading of the interference colors. Throughout this period the diffusion of oxygen into the metal takes place; at 300 C, however, extrapolation of Pemsler's data[12] indicates that this is very slow and may be neglected in considering the change in rate law.

The change in appearance of the oxide film corresponds roughly with the region in which the diffusion of oxygen up to the oxide/metal interface via defects in the oxide layer becomes significant compared with that diffusing via anion vacancies in the film; thereafter the diffusion along these easier diffusion paths becomes rate determining. In the case of arc-melted zirconium a ready path is presented along the grain boundaries and growth continues at a rate which is still approximately parabolic. However, since the majority of the oxygen is now diffusing along grain boundaries, oxide film growth is restricted to narrow bands along these boundaries. This results in stresses tending to extend the oxide in these regions in opposition to the compressive stress in the thin continuous film resulting from the high Pilling-Bedworth ratio of 1.56.[13] This results in cracking of the oxide and more rapid film growth ensues via these cracks producing further cracking and ultimate failure of the film within the grains as a result of a stress relieving process.

Zircaloy-2, however, changes uniformly from the interference color film to the dark oxide and no evidence of non-uniform growth of oxide has been obtained. This implies that there are sufficient diffusion paths available within the protective film to enable uniform film growth to continue when the thin film has reached its limiting thickness. Enhanced diffusion along grain boundaries would be expected to occur in this case also and the provision of diffusion paths in the body of the oxide film to counterbalance this may be the result of the distribution of the alloying elements. On the other hand the alloying elements may operate by preventing, or reducing, the grain boundary diffusion. There is no positive evidence at present as to the positions of the alloying elements in Zircaloy-2 and work is in progress here to determine this.

It appears that the critical point in the corrosion of arc-melted zirconium occurs at a very early stage in the film growth when diffusion along grain boundaries supercedes diffusion via anion vacancies as the principal route for oxygen access to the metal. Subsequent non-uniform film growth results in mechanical cracking of the oxide and eventual complete failure of the film. Zircaloy-2 satisfactorily survives this change and uniform film growth throughout results in the formation of the thick protective film which subsequently fails at a later period (the "transition" point).

Conclusion

Oxide film breakdown in arc-melted sponge zirconium occurs as a result of cracking along the grain boundaries. This is the product of enhanced film growth in a narrow band along selected grain boundaries, combined with the effect of variations in oxidation rate with grain orientation when these are large. The severity of this attack and the ensuing cracking is a function of the misorientation of the two grains at the boundary. This enhanced film growth at the boundaries has not been observed to any significant extent during the early stages of oxidation but becomes apparent at the stage when the interference color film gives way to the thicker dark oxide. The darkening of the oxide becomes apparent first along the grain boundaries in this material whereas in arc-melted Zircaloy-2 uniform darkening of the film is observed. This darkening in the film is thought to be due to the progressive increase in the vacancy gradient in the oxide during oxidation leading to a black non-stoichiometric oxide. The onset of non-uniform film growth is thought to be related to the point where the amount of oxygen supplied to the metal surface via grain boundary diffusion and other easy diffusion paths exceeds that supplied by diffusion through the uniform film via anion vacancies.

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Ablation Behavior of Materials Subjected To Missile Re-Entry Heat Flux Rates*

By R. M. KRUPKA and D. E. TAYLOR

Abstract

Hypersonic vehicles, during re-entry, are exposed to transient heating involving extremely high heat flux rates. These heat flux rates have been simulated in the laboratory with the plasma discharge from high-intensity, air-stabilized electric arcs of approximately 1000-kw power rating. This paper describes the behavior of samples of ten different materials tested in this facility, including four metals, three ceramics, two plastics, and graphite. Ablation rates, heat balances, and effective heats of ablation are presented. The influence of changes in the test environment upon material behavior is indicated. In the highest temperature test environment utilized, the plastic and graphite samples provided lower stagnation point weight loss rates during ablation than the metal and ceramic samples.
3.5.9

Introduction

THE OUTER surfaces of hypersonic vehicles are subjected to severe transient thermal environments during the re-entry phase. In these environments, the stagnation temperature of the gases flowing about the vehicle become much higher than the maximum surface temperatures of the skin material. The associated high heat flux rates for a vehicle in the ballistic missile category prevent intact survival of materials in most cases. Some ablation usually occurs.

The study of material behavior under these conditions is a complex problem. In many cases, mathematical study of the degradation process for specific materials in the actual environments becomes tenable only after testing under simulated conditions reveals the important physical transport and protective phenomena involved during degradation. Heat flux rates and time durations of interest may be simulated by exposing materials to the plasma discharge from high-intensity, air-stabilized electric arcs. Test results obtained for ten different materials are presented in this paper.

Experimental Procedure

Description of Equipment

The Laboratories for Applied Sciences (University of Chicago) has been engaged in the development of stabilized arc equipment for testing purposes since July 1956. Units, designed to operate with various stabilizing mediums, have been built with several power ratings from 80 kw to 3000 kw. The arc used in the tests reported here was designed for nominal operation in the 1000-kw range and uses air as the stabilizing medium. A schematic diagram of the device is shown in Figure 1. The arc uses a 1½ inch graphite anode and a graphite cathode with a 1¼ inch orifice. Air enters the arc chamber tangentially to provide sufficient swirl for a stable air column within the chamber. More detailed descriptions of the equipment are given in Reference 1.

Operating Conditions

For material testing, it was desired to expose the samples to a nominal heat flux rate to the stagnation point of a cold non-ablating surface of approximately 2000 Btu/ft²-sec. It was determined experimentally that this requirement could be met by operating the arcs and positioning the samples in accordance with the conditions described in Table 1.

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Samples Tested

This paper presents test results obtained for copper, aluminum, stainless steel, molybdenum, zircon, mullite, alumina, linen bakelite, nylon, and graphite samples.

The sample shape for all materials, except nylon and molybdenum, is shown in Figure 2. The nylon sample was shortened to allow manufacture from a 3-inch diameter bar. In the case of molybdenum, a 2-inch diameter bar was utilized. A full-size cone was fabricated by skirting the base with stainless steel. The ceramic samples were cast into the proper shape; all other materials were machined from bar stock.

Test Procedure

The sample was mounted vertically 22 inches above the cathode in the specimen holder. It was protected from the plasma discharge by a shield. After the arc operating conditions listed in Table 1 were obtained, the sample and the shield were lowered until the sample was in the proper position. At this time, the shield was withdrawn to expose the sample. This sudden exposure yielded a step change in the heat input.

Information obtained during the tests included color motion pictures taken at 32 frames per second. These motion pictures allowed the construction of the curves of stagnation point ablation versus time for the test period (shown in Figure 3). Some still photographs were also taken during the tests; three examples of these photographs are shown in Figure 4.

Analysis of Results

The experimental results were correlated by obtaining:

1. Transient heating analysis for the period before melting of the surface occurred.
2. Heat balances for the first second of ablation.
3. Effective heats of ablation based upon the maximum observed rates of ablation.

The salient features of each of these computations and the results obtained are presented below.

Transient Heating Before Melting

Examination of the motion pictures of the tests showed that isothermal surfaces in the metal samples in the vicinity of the axis of symmetry were approximately plane and perpendicular to the axis. This justified the use of one-dimensional heat flow analyses. During the period prior to melting, it was assumed that the samples were convectively heated, with heat flow perpendicular to the sample surfaces at the axisymmetric stagnation point, and that the samples were of such length that they could be regarded as equivalent to a semi-infinite solid. This allowed use of well-known analytical soultions for the temperature distribution within the body. In particular, the temperature distribution at the instant melting starts is given by

$$\frac{T(x,t) - T_o}{T_g - T_o} = \operatorname{erfc}\frac{z}{2} - \exp(Nz + N^2)\operatorname{erfc}\left(\frac{z}{2} + N\right) \quad (1)$$

where $z = \dfrac{x}{\sqrt{\alpha t}}$

$$N = \frac{h}{k}\sqrt{\alpha t}$$

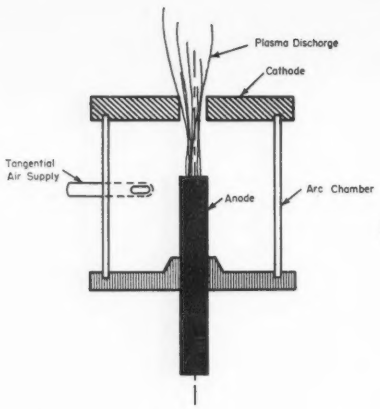


Figure 1—Schematic diagram of air stabilized arc.

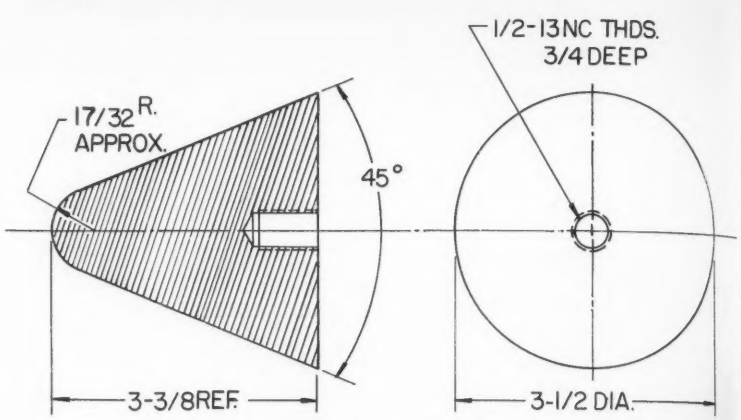


Figure 2—Standard sample shape.

TABLE 1—Operating Conditions

Property	Value
Arc current, amp	3500
Arc voltage	300
Air flow, lb/sec	0.25
Heat flux rate, *Btu/ft²-sec	1950
Distance of sample stagnation point above cathode, in.	4.5
Plasma temperature at stagnation point, Degrees K	8000-9000
Plasma velocity, ft/sec	2500

*As determined by fusible copper calorimeters.

and T (x, t) = temperature at a distance x along the sample axis from the surface at the instant melting starts
T_o = initial temperature of sample at time zero
T_g = temperature of gas stream
α = thermal diffusivity of sample material
t = time of exposure at which melting starts
h = film coefficient of heat transfer
k = thermal conductivity of sample material.

At the sample surface, Eq. (1) takes the special form

$$\frac{T_m - T_o}{T_g - T_o} = 1 - \exp N^2 \operatorname{erfc} N \qquad (2)$$

where T_m = melting temperature of the sample material. Equation (2) was used in the following manner. The gas stream temperature was estimated as approximately 8500 K from spectroscopic measurements. For the metal samples, the times to reach melting temperature were obtained from the motion pictures, and Eq. (2) was used to determine the associated value of h, the film coefficient of heat transfer. This value of h was then used in Eq. (1) to determine the temperature distribution at the instant melting started. For the ceramic and graphite samples, the times to reach melting temperature were difficult to establish from the motion pictures. Therefore, an average value of h was assigned for these materials; from this, the times to reach melting temperature and the temperature distributions at the melting times were computed. The linen bakelite and nylon samples were not analyzed in this manner, since their behavior was not primarily dependent upon conditions within the original solid material.

Numerical values used in conjunction with Eqs. (1) and (2) and the derived values of either the film coefficient or the melting time are shown in Table 2. Two sets of results are included for the graphite sample, one set corresponding to sublimation at 6600 F and the other set to combustion to carbon monoxide at 5480 F (3300 K).

Heat Balances for the First Second of Ablation

With the aid of the information obtained above, heat balances were computed for the first second of ablation. In these heat balances, it was assumed that liquids formed due to melting were immediately swept away, and their resistance to heat transfer was neglected. The basic heat balance used was

$$q_{convected} + q_{chemical} = q_{phase\ change} + q_{radiated} + q_{sensible} \qquad (3)$$

where $q_{convected} = h (T_g - T_m)$
$q_{phase\ change} = \rho L \dot{s}$
$q_{radiated} = \varepsilon \sigma T_m^4$

and

q = heat transferred per unit area in first second of ablation
ρ = specific weight of sample material
L = latent heat of fusion or sublimation of sample material
$\dot{s}$ = initial linear ablation rate
ε = emissivity of surface of sample material
σ = Stefan-Boltzmann constant.

The chemical heating term was specifically computed only for the graphite sample in a manner similar to that used for the phase change term; the effect of chemical reactions on other samples was absorbed in the value of the film coefficient, h, employed.

Evaluation of the sensible heating term in the heat balance involves determination of the transient temperature distribution within an ablating body. This is a complex problem; discussions of the analytical difficulties involved are contained in References 1 and 2. Results of sufficient accuracy for the heat balance comparisons were obtained from approximate graphical solutions. From the motion pictures, the ablation displacement s (t_1) corresponding to one second of ablation was obtained. The temperature distribution at the instant melting begins was obtained from Eq. (1). It was assumed that the temperature at x = s (t_1) changed linearly with time from the value given by Eq. (1) to the ablation temperature, T_m, during the first second of ablation. With this assumption, the internal temperature distribution after one second of ablation could be determined from standard graphical techniques. The sensible heating term was then found from

$$q_{sensible} = \rho c_p \left\{ \int_0^{s(t_1)} [T_m - T(x,t)]\,dx + \int_{s(t_1)}^{\infty} [T(x,t_1) - T(x,t)]\,dx \right\} \qquad (4)$$

where c_p = specific heat of sample material.

Numerical values employed in the heat balance computations are shown in Table 2.

The results obtained are represented in the bar graphs shown in Figure 5. Two bars are shown for each material tested. In one bar, the heat furnished to the sample by convection and combustion are added together; in the other bar, the heat utilized by the sample due to sensible heating, phase changes, and radiation are added together.

In the absence of unusual phenomena, the two bars should be of comparable height. Two heat balances are shown for the graphite sample, one assuming sublimation at 6600 F and the other combustion to carbon monoxide at 5480 F.

Effective Heats of Ablation

The effective heat of ablation is a figure of merit commonly applied to comparing the effectiveness of different ablating materials in withstanding high tem-

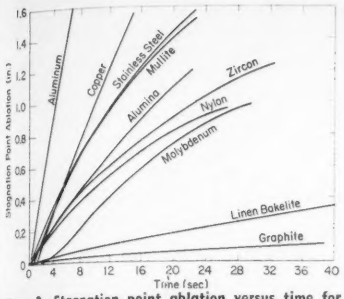
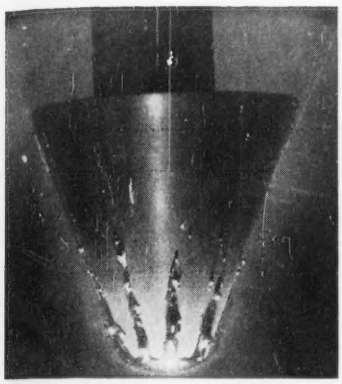
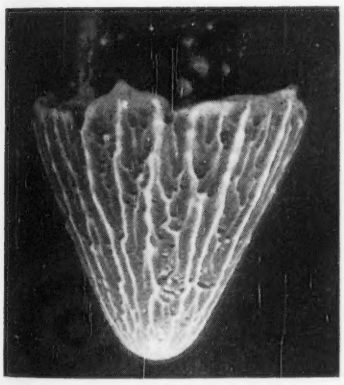


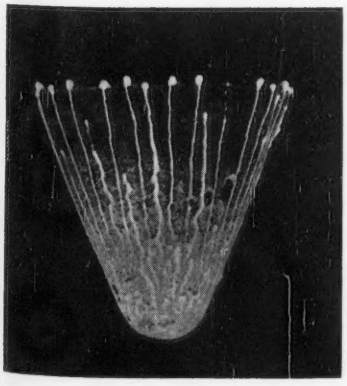
Figure 3—Stagnation point ablation versus time for materials tested.



Copper



Mullite



Zircon

Figure 4—Three samples during initial part of test period.

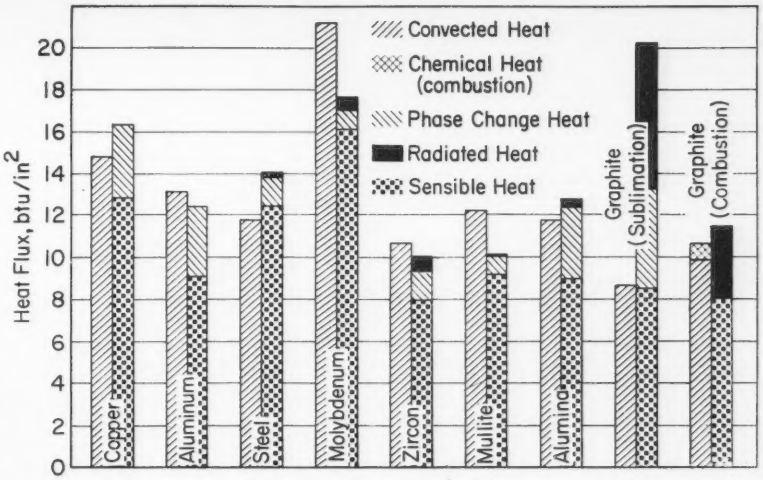


Figure 5—Heat balance results.

perature environments. It may be defined as the quotient of the convective heat transfer to a non-ablating body divided by the rate of weight loss from the ablating surface. Numerical values of this quantity were obtained for the samples tested, based upon the maximum observed rates of ablation at the stagnation point. The effective heats of ablation presented were therefore determined from the expression

$$H_{eff} = \frac{h\,(T_g - T_m)}{\rho\,\dot{s}_m} \qquad (5)$$

where H_{eff} = effective heat of ablation of sample material

$\dot{s}_m$ = maximum observed rate of linear ablation.

The film coefficients used in Eq. (5) were those given in Table 2. The average film coefficient used for the ceramic and graphite samples was also used in computations for the linen bakelite and nylon samples, as well as the following data:

	Specific Weight (lb/in^3)	Assumed Surface Temperature (°F)
	ρ	T_m
Linen Bakelite	0.0473	5000
Nylon	0.0404	600

The results of the computations for all samples tested are given in Table 3.

Discussion of Results

The heat balance results presented in Figure 5 are based on published, rather than measured, values of physical constants for the materials tested and on several simplifying analytical assumptions. Consequently, without specific cause, differences of the order of 10 percent between the computed values of the heat furnished and heat utilized by individual samples may be expected.

Results for Metal Samples

The heat balance results for the copper and aluminum samples agree to within tolerable errors.

For the steel specimen, the computed heat utilized exceeds the heat furnished by 16 percent. It is believed this discrepency is due to oxidation, which appeared to start on this specimen almost at the instant melting occurs. As a consequence, the heat actually furnished would be greater than that computed on the basis of the effective convective film coefficient prior to melting.

The computed heat furnished to the molybdenum sample exceeds the heat utilized by 20 percent. Motion pictures reveal that several phenomena occurred during the test of this sample. Prior to melting of the molybdenum proper, a thin liquid film was observed on the forward portion of the sample. Simultaneously, gas was evolved from the surface. The data contained in Table 2 also demonstrate that the film coefficient of heat transfer, based on the time to melt, was much higher for the molybdenum sample than for the other metal samples. All of this evidence indicates that oxidation reactions were present during most of the period prior to melting.

The observed behavior in the motion pictures suggests the formation of MoO_3, which melts at 1460 F and evaporates to a gas at 2100 F at atmospheric pressure. It is possible that the rate of oxide formation may have changed after melting of the molybdenum proper occurred. If so, this may account for the deviation in the heat balance results. Also, the surface temperature of the molybdenum during melting is high enough so that accurate determination of the contribution of radiation to the total heat utilized is important in the heat balance results. It is possible that the emissivity of the sample surface was actually higher than that indicated in Table 2.

The effective heats of ablation listed for the metal samples in Table 3 are based upon the maximum rate of ablation observed during the tests and the initial heat flux rate at the beginning of ablation. In some cases, the computed temperature distribution within the body at the beginning of ablation differs widely from that corresponding to conditions of

TABLE 2—Numerical Values Used in Computations

Material	Specific Weight lb/in³ ρ	Specific Heat Btu/lb-°F c_p	Thermal Conductivity Btu/in-°F-sec k	Thermal Diffusivity in²-sec α	Latent Heat Btu/lb L	Melting Temperature °F T_m	Emissivity ε	Time to Melt sec t	Initial Ablation Rate in/sec $\dot{s}$	Film Coefficient Btu/in²-sec-°F h^1
Copper	0.324	0.092	0.00526	0.176	91	1981	0.60	1.9[2]	0.115	0.00114[5]
Aluminum	0.0975	0.215	0.00297	0.141	138	1220	0.19	0.35[2]	0.246	0.000952[5]
Stainless Steel	0.283	0.15	0.000328	0.0077	50	2890	0.60	0.57[2]	0.096	0.000976[5]
Molybdenum	0.369	0.061	0.00196	0.087	124	4760	0.24	1.4[3]	0.020	0.00208[5]
Zircon ($ZrO_2 \cdot SiO_2$)	0.170	0.131	0.000091	0.0041	146	4622	0.30	0.23[4]	0.0571	0.00104[5]
Mullite ($3Al_2O_3 \cdot 2SiO_2$)	0.114	0.23	0.000116	0.0044	82	3166	0.30	0.13[4]	0.0875	0.00104[5]
Alumina (Al_2O_3)	0.135	0.271	0.000144	0.0039	459	3630	0.30	0.33[4]	0.0556	0.00104[5]
Graphite	0.059	0.47	0.000465	0.0167	{25,200 ~4,300}	{6600 5480}	0.83	4.35[4] 2.52[4]	0.0032	0.00104[5]

[1] Plasma temperature estimated at 8500 K.
[2] Observed from motion pictures.
[3] Calculated from observed time to melt.
[4] Calculated from thermal properties and average film coefficient.
[5] Average film coefficient assumed for these materials.

TABLE 3—Effective Heats of Ablation

Material	Maximum Ablation Rate, in/sec. $\dot{s}_m$	Effective Heat of Ablation, Btu/lb H_{eff}
Copper	0.115	397
Aluminum	0.246	544
Stainless Steel	0.096	433
Molybdenum	0.055	1,045
Zircon	0.0571	1,110
Mullite	0.0875	1,225
Alumina	0.0556	1,570
Linen Bakelite	0.0130	16,800
Nylon	0.0470	7,850
Graphite	0.0032	52,000

TABLE 4—Effect of Thermal Environment on Effective Heat of Ablation

Distance of Sample Stagnation Point Above Arc Cathode	Plasma Temperature at Stagnation Point, °K	Effective Heat of Ablation, Btu/lb	
		Nylon	Teflon
4.5	8000-9000	8830	9350
9.0	4000	1945	2550

steady-state melting. This delays the establishment of steady-state ablation conditions usually until the initial heat flux rate has been markedly reduced by either increase in the distance between the arc cathode and the sample stagnation point or by increased bluntness of the stagnation region. For these cases, the effective heats of ablation given in Table 3 will be in excess of those which would be obtained under steady-state heating conditions identical to those which existed at the beginning of ablation. This effect is of significant importance on the copper and molybdenum samples; true values for these materials for the nominal test conditions are probably of the order of 350 and 700 Btu/lb, respectively.

Results for the Ceramic Samples

All of the ceramic samples achieved some protection from liquid films of the molten material, which acted as thermal insulating layers between the hot gases and the solid surface. This protective action was offset to varying degrees on the different samples by the loss of material by means of a flaking action, evidently caused by failure due to thermal stresses. This was an important process in determination of the total degradation rate of the samples, since some fraction of the material was stripped away prior to providing the protection afforded by heat absorption due to both sensible heating to the melting point and the latent heat of fusion. The flaking action was the least noticeable on the zircon sample and the most severe on the alumina sample. The severe flaking on the alumina sample was especially detrimental because of the high latent heat of this material. This phenomenon evidently accounts for the fact that, in the heat balance shown in Figure 5, the computed heat utilized by the alumina sample exceeds the heat furnished. The computation of the heat utilized assumed that all of the material ablated was heated to the melting point and then changed phase; this was not actually the case, since a significant fraction of the material flaked away.

The effective heats of ablation listed in Table 3 for the three ceramic samples are not significantly affected by the transient temperature distribution effect discussed previously for the metal samples.

Results for the Graphite Sample

Two heat balances were computed for the graphite sample: one assuming that sublimation took place at 6600 F, the other assuming that combustion to carbon monoxide took place without sublimation at a temperature of 5480 F, which was a surface temperature observed with an optical pyrometer. It is apparent from the results shown in Figure 5 that the energy balance is drastically improved for the case assuming combustion; hence this must have been the ablation mechanism for this sample.

The effective heat of ablation given in Table 3 for the graphite sample is significantly affected by the transient temperature distribution effect discussed previously. In addition, the rate of mass loss from a sample re-radiating a large fraction of the incident energy becomes extremely sensitive to small changes in the surface temperature and/or to changes in the incident heating caused by changes in bluntness of the sample. Silhouettes of this sample before and after testing indicate that the sample was inadvertently fabricated with excessive initial bluntness in the stagnation region. For these reasons, the effective enthalpy given in Table 3 is believed to be much greater than that which would actually be obtained for steady-state heating at the nominal test conditions.

Results for the Plastic Samples

The plastic materials tested represent examples of materials which decompose at relatively low temperatures and discharge large amounts of gaseous products into the boundary layer. For such materials, cooling by mass transfer action predominates; the mathematical model employed to construct the heat balances shown in Figure 5 would not provide an adequate explanation of material behavior. The presentation of results for the plastics has therefore been restricted to the effective heats of ablation given in Table 3.

The behavior of the linen bakelite sample was typical of that provided by certain classes of plastics tested in the arc facility. A cokelike layer was formed upon heating; this furnished protection for the material beneath it by providing thermal insulation and a high temperature reradiating surface. The density of this layer was considerably less than that of the original solid material. In computing the results shown in Table 3, the thickness of the coke-like layer (as determined by sectioning of the sample) was regarded as ablated material, and the linear ablation rate was determined accordingly.

During the test of the nylon sample, a flow disturbance was observed near where the spherical tip joined the straight part of the cone. The ablation rate greatly increased in this region, causing a neck to form.

The values listed in Table 3 for the plastic samples are representative of steady-state values for the imposed test conditions, but they should not be regarded as unique for a given material. The effective heat of ablation for any material which is significantly protected by gaseous mass transfer action is dependent upon the characteristics of both the material and the thermal environment to which it is exposed (see References 3 and 4). In particular, theory indicates it

will be directly proportional to the temperature or enthalpy difference across the boundary layer which acts as a driving force for heat transfer. This phenomenon has been verified experimentally at the Laboratories for Applied Sciences in a series of tests where the effective heats of ablation of nylon and Teflon were determined by somewhat different methods at two test positions in the arc discharge (see References 5 and 6). The results of these tests, which are summarized in Table 4, clearly indicate the strong dependence of the effective heat of ablation on the temperature difference causing heat transfer. Additional discussion of the performance of various plastics when tested in the arc discharge under the conditions listed in Table 1 are contained in Reference 7.

Conclusions

The information obtained from the tests described warrants the following conclusions:

1. Laboratory testing aids in determination of important phenomena involved in the ablation process for a given material. This is exemplified by the observation of chemical reactions, liquid films, loss of material due to thermal shock, and the formation of carbonized shells. This type of information is of great value in the formulation of mathematical models which will serve to adequately predict the material behavior in intended applications.

2. In environments where materials are exposed to temperatures higher than the melting temperatures of refractory metals or oxides, certain plastics may provide the best thermal resistance to the environment for either short-time or continuous duty.

3. The performance of materials which offer significant thermal resistance due to mass transfer action is closely related to environmental conditions to which the materials are exposed. In particular, the performance of all materials, as measured by the effective heat of ablation, will ordinarily increase as the temperature of the environment increases. However, the relative rating of different materials may change with temperature or other environmental conditions. For this reason, it is impossible to arrive at ratings of materials for severe thermal environments from the results of tests at a single environmental condition. In addition, if tests are to be used to aid in selection of materials for high temperature service in specific applications, the test environments should simulate as closely as possible the conditions of the actual environment.

Acknowledgments

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* Chicago Midway Laboratories is now a division of the Laboratories for Applied Sciences of The University of Chicago.

Any discussion of this article not published above will appear in December, 1960 issue.

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Cathodic Protection For Metallic Water Tanks*

By CONRAD A. BAUER and LEVI C. EDDY

Introduction

CATHODICALLY protecting the inside of water tanks against corrosion is becoming a common practice. This is especially true for the large tanks used by municipalities and industries for storing water. Many of these cathodic protection systems are installed by "rule of thumb" tempered with past experience.

Some years ago the authors were asked to apply cathodic protection to a large potable water tank which management felt was costing too much to keep painted. Since there was no past experience to draw on it was necessary to lay out a system for cathodic protection based on theory. In like manner additional systems have since been installed. All are giving the desired protection as evidenced by semi-annual inspections.

It is felt that others, particularly engineers with no previous experience, would be interested in the approach used here.

Theory of Current Distribution

Consider two anodes "A" and "B," (see Figure 1), placed parallel to each other a distance d_t apart in a uniform solution of infinite area and uniform depth. The anodes are long enough so that end effects may be neglected. If these two anodes are connected to an electrical potential of E volts, a current $I = E/R_t$ amperes will flow. The current "I" will be composed of an infinite number of parallel currents "i_1" following the paths represented by the dotted lines. Since the lengths of the separate paths are different, the resistance "R" of each path will be proportional to its length and therefore the current "i_1" will be inversely proportional to the length of its path. That is i_a will be greater than i_b.

If a test probe is put into the solution and connected to Anode "A" through a voltmeter it will measure the voltage drop "e_1" along a current path passing the test probe. Moving the test probe so that "e_1" remains constant will cause it to trace a circle eccentric to Anode "A," crossing all the current paths at right angle as shown by the solid lines "e_1" . . . "e_n." This then is an isopotential circle.

Placing a conducting metal cylinder of the identical diameter as the isopotential E_1, and coinciding with its location, will not change the magnitude of the current or its pattern of flow. Let this cylinder now be a water tank with Anode "A" in its liquid, and let "E" be the rectifier voltage between the anode and the tank which will cause current "I" to flow. To determine the above mentioned current pattern within a tank it is necessary to locate an imaginary anode "B" as the image of anode "A" with respect to an imaginary neutral line outside the tank. This will be explained under the heading of Anode Size, Appendix 1. As anode "A" approaches the center of the tank, "B" approaches the outside of the tank, becoming the tank when "A" becomes concentric with the tank.

Basic Factors

Before starting any calculations it is necessary to establish the contributing basic factors affecting the design.

1. Aggressiveness of the liquid varies from one source to another and requires different values of impressed current to afford protection. Most well waters in the middle west will require from 0.5 to 10 milliamperes per square foot of tank surface for protection. Use 7 milliamperes for design purposes, unless information to the contrary is available, to assure a rectifier of adequate capacity.

2. Resistivity of the water "ρ" depends on its pH value and total solids content. Instruments are available to measure the water resistivity—usually in ohms per square centimeter per centimeter at 75 F. ρ may vary from 1100 to 4000 but for any one source it may be taken as a constant. If no test data are available, use $\rho = 4000$, in calculating anode resistance, so that sufficient anodes will be installed and redesigning thus avoided.

3. Anode material used will depend on whether or not the water is exposed to freezing. If the water may freeze and thus break the anodes or the suspension, then the less expensive aluminum anodes are used and replaced each spring. If the water is not likely to freeze, then the more expensive high silicon iron anodes, that require less maintenance and last longer, are preferred.

4. Rectifier voltage determines the power costs. Increasing the voltage will increase the power consumed but may reduce the first cost since fewer anodes will be required. As a first try select a rectifier voltage $E = 2 + D_t/3$ volts, where D_t is the tank diameter in feet. After the type, size, number and location of the anodes have been decided on, then $E = E_c + (\pi D_t i r_t)/F$. See appendix 5.

5. Current required is dependent on the nature of the liquid and the square foot of surface area to be protected, and is independent of the voltage.

$$I_t = i\,[\pi D_t (h_t - D_t/4) + A]\ 10^{-3}$$

Appendix 5.

Bauer

Eddy

CONRAD A. BAUER received a BS in electrical engineering from the Milwaukee School of Engineering (1924). He was employed by the Commonwealth Edison Co. from 1924 until 1960, retiring as Supervising Line Design Engineer in charge of the Line rating and Temperature Control Group and the Corrosion and Electrolysis Control Group. He is a Professional Engineer (Illinois).

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Abstract

Practical and theoretical considerations are detailed in connection with the cathodic protection of a large potable water tank. Topics discussed include theory of current distribution on parallel anodes, aggressiveness and resistivity of water, size and spacing of anodes, anode interference factor, and rectifier voltage and current requirements. A large number of equations used in calculating circuit resistances, current requirements, outputs, etc. are reported.

5.2.1

A = Area in square feet of submerged surfaces other than the tank.

Calculations

1. **Number and size of anodes.** As a first approximation assume an anode located in the center of the tank. Assume further that a wafer one foot high has been sliced through the tank. Then the resistance

$$r = E/I_a \text{ ohms for this section.} \qquad 3\text{-}1$$

Also,

$$r = 0.00522\,\rho L_a\,(D_t/d_t), \text{ therefore} \qquad 3\text{-}2$$

$$d_t = D_t/e^k, \text{ anode diameter in ft.} \qquad 3\text{-}3$$

$$\text{where } k = (7.33\ E\ 10^{-5})/(i\rho D_t) \qquad 3\text{-}4$$

★ Submitted for publication August 17, 1959.

TABLE 1—Cathodic Protection for Water Tanks

Material	E Volts	S	K_e	P	Q for $\xi = 1.0$	W for $\xi = 1.0$	ξ
Calcium	—2.87	1.55	X10⁻⁴	.....	.....	.....	.....
Sodium	—2.712	0.97	.....	.....	.....	.....	.....
Magnesium[1]	—2.34	1.74	1.260	0.592	1000	8.76	0.4—0.6
Aluminum	—1.67	2.71	0.936	0.922	1345	6.5	0.4—0.6
Zinc[2]	—0.762	7.14	3.387	2.43	372	23.6	0.9
Bright Steel	—0.55	7.7	.....	.....	.....	.....	.....
Iron	—0.44	7.85	2.893	2.68	435	20.1	1.0
Nickel	—0.25	8.77	3.040	2.98	415	21.1	.....
Tin[3]	—0.136	6.6	.....	2.24	.....	42.9	.....
Cast Iron	—0.27	7.03	.....	2.39	.....	.....	.....
Lead	—0.126	11.34	10.731	3.86	117.1	74.6	.....
Rusty Iron	—0.06	7.5	.....	2.55	.....	.....	.....
Hydrogen	0.0	.....	0.105	.....	.....	.....	.....
Copper (cupric)	+0.345	8.91	3.3	3.04	382	22.9	.....
Copper Sulfate	+0.34	3.52	.....	.....	.....	.....	.....
Copper (cuprous)	+0.522	8.91	6.59	3.04	191	45.7	.....
Carbon "Graphite"[4]	+0.73	3.51	0.317	1.195	3970	2.2	.....
Platinum	+1.2	21.37	10.104	7.26	124.5	70.5	.....

E = potential in volts with respect to hydrogen
K_e = electrochemical equivalent, grams per coulomb
P = weight per inch foot of rod = 0.34S, lbs.
Q = ampere hours per lb = $0.126/(K_e\xi)$
S = specific gravity
W = weight loss per ampere year = K_e (seconds in one year/grams per lb.) = 69,500 K_e
ξ = efficiency of material in decimals; increases with current density.

[1] Magnesium ribbon in ducts must be moved periodically, and is considered no longer effective after 20 percent is consumed.

[2] Zinc anodes become polarized except in salt water areas. At 185° zinc is cathodic to iron.

[3] In the absence of air tin becomes anodic to iron in most acid solutions.

[4] Limit carbon anodes to one ampere or less per square foot of anode surface. Life of anode is about 15 years if backfill furnished is used. Life of anode is about 4 years if no backfill is used.

The anode diameter "d_1," may be impracticable. If so it can be replaced with "N" smaller anodes forming a ring of diameter "d_1."

The minimum number "N" of uniformly spaced anodes required to give even coverage is,

$$N = \pi D_1/(D_1 - d_1) \qquad 3\text{-}5$$

The distance "L" from the anode to the nearest surface of the tank is,

$$L = 0.5(D - d), \text{ inches} \qquad 3\text{-}6$$

If there are obstructions such as ladders, overflow and/or intake pipes, sand traps and the like, the number of anodes may have to be increased and changed from the ring calculated above, so as to get uniform current distribution. This arrangement can also be calculated but more often is done by spotting them on a plan view of the tank and its obstructions with the above mentioned anode ring superimposed. After making these corrections the size of the individual anode can be calculated for the new resistance value

$$r_1 = NEF/I_o, \text{ ohms per ft. of anode.} \qquad 3\text{-}7$$

E = voltage of rectifier
F = interference factor (see appendix 3)
I_o = current per ft. height of tank = $\pi D_1 i \times 10^{-3}$
D_1 = tank diameter in feet.
i = current required per square ft. of surface, milliamperes
N = number of anodes (in outer ring if more than one ring of anodes are installed)

Anode Size (see appendix 1)

Based on permissible resistance

$$d_a = 2L(D - L)/Re^k \text{ inches} \qquad 4\text{-}1$$

$$k = NFr/K \qquad 4\text{-}2$$

D, L and R in inches.

Based on material consumed per year

$$d_b = \sqrt{53.5\ iDK_e/(NS\xi)} \text{ inches} \qquad 4\text{-}3$$

$d = d_a$ or d_b whichever is the larger

Anode height above tank bottom (see appendix 2)

$$h_2 = (d/2)\ (1 + \sqrt{1 + 0.2252\ (L^2/d)\ \log_e [2L(2R - L)/dR]}), \text{ inches} \qquad 4\text{-}4$$

Stub Anodes (see appendix 2)

$l_s = 0.262L$ inches, length of anode 4-5

d = same as for side anodes.

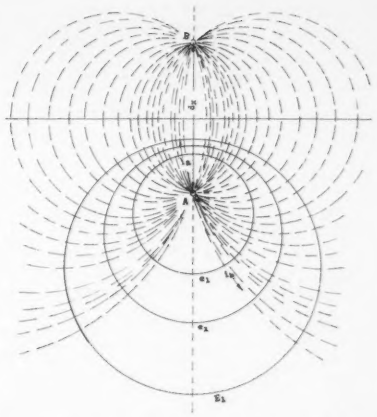

Figure 1—Field for two parallel anodes placed a distance d_x apart in a uniform solution of infinite area and uniform depth.

$$N_s = \pi R(R - 2L)^2/4L^2(R - L), \text{ number of Stub Anodes.} \qquad 4\text{-}6$$

Anode Interference Factor (see appendix 3)

$$F = [G - (g_2 + \ldots + g_n)]/G \qquad 4\text{-}7$$

Where

G = geometric factor of reference anode (anode No. 1)

g = geometric factor of isopotential from anode No. 1 passing through anode No. 2, and so on through each anode to and including anode n.

Current Resistance (see appendix 4)

The total circuit resistance per foot of anode consists of the resistance through the liquid, the resistance through the deposit on the anode and the metal to liquid contact resistance at the anode and at the tank, that is,

$$r = r_l + r_a + r_c \text{ ohms.} \qquad 4\text{-}8$$

where

r_l = resistance through liquid for one anode to tank
r_a = resistance through deposit on the anode
r_c = resistance of metal to liquid and liquid to metal surfaces at the anode and at the tank.

The total resistance per foot cross section of tank r_t = the reciprocal of the sum of the reciprocals of the resistance for all anodes.

Rectifier Voltage required. (see appendix 5)

$$E = E_c + [(\pi D_1 i r_t)/F]\ 10^{-3} \text{ volts} \qquad 5\text{-}1$$

If the anodes are placed symmetrically then

$$E = E_c + [(\pi D_1 i r)/(NF)]\ 10^{-3} \text{ volts} \qquad 5\text{-}2$$

Rectifier Current required. (see appendix 5)

$$I_r = i\pi D_1(h_1 + D_1/4)\ 10^{-3} \text{ amperes} \qquad 5\text{-}3$$

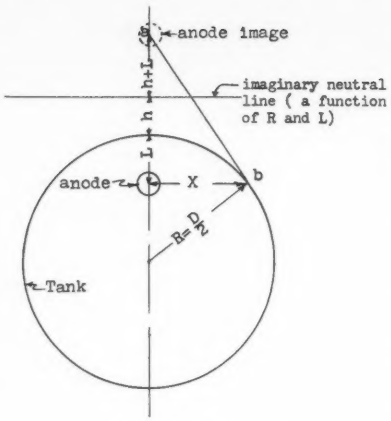

Figure for Appendix 1

APPENDIX NO. 1

Anode Size

Anode size based on permissible resistance per anode.

$\angle abo = 90°$

All dimensions in inches

$$\frac{X}{R-L} = \frac{2(h+L)}{X} \qquad 6\text{-}1$$

$$X^2 = 2(h+L)\,(R-L) \qquad 6\text{-}2$$

$$R^2 = X^2 + (R-L)^2 = 2(h+L)\,(R-L) + (R-L)^2 \qquad 6\text{-}3$$

$$h = L^2/2(R-L) \qquad 6\text{-}4$$

$$h+L = [L^2 + 2L(R-L)]/2(R-L) \qquad 6\text{-}5$$

$$= L(2R-L)/[2(R-L)] \qquad 6\text{-}6$$

$$h+R = [L^2 + 2R(R-L)]/[2(R-L)] \qquad 6\text{-}7$$

$$= [R^2 + (R-L)^2]/[2(R-L)] \qquad 6\text{-}8$$

Resistance Calculation

$$K = 0.00522\,\rho \qquad 6\text{-}9$$

$$r_2 = L \log_e 4(h+L)/d_a = K \log_e \{2L(D-L)/[d_a(R-L]\} \text{ anode to } \pm \qquad 6\text{-}10$$

$$r_3 = K\,(\log_e (2h+L)/L = K \log_e R/(R-L) \text{ tank to } \pm \qquad 6\text{-}11$$

Resistance, anode to tank

$$r_1 = r_2 - r_3 = K[\log_e 2L(D-L)/\{d_a(R-L)\} - \log_e R/(R-L)] \qquad 6\text{-}2$$

$$= K \log_e 2L(D-L)/d_aR \text{ ohms per ft. of anode} \qquad 6\text{-}13$$

Permissible Resistance per Foot of Rod

$r_1 = NFr$, ohms (see equation 3-5 for N, 6-13 for r, and 9-9 for F) 6-14

Required diameter of anode based on permissible resistance, equating 6-13 and 6-14

$$d_a = 2L(D-L)/(Re^{k_2}) \text{ inches} \qquad 6\text{-}15$$

$$k_2 = NFr/K \qquad 6\text{-}16$$

Required diameter based on material consumed per year, Efficiency $= \xi$

$$d_b = \sqrt{iCW(1000\,NP\xi)} = \sqrt{53.5\,i\,DK_e/(NS\xi)} \text{ inches} \qquad 6\text{-}17$$

(See Table 1 for values of C, W, P, K_e, S and ξ.)

$C = \pi D_1$ Circumference of tank in feet.

Anode diameter $d = d_a$ or d_b, whichever is the larger.

APPENDIX NO. 2

Anode Height Above Tank Bottom

The anode should be far enough from the tank bottom so as not to concentrate a current leakage from the anode tip. It is recognized that the anode tip can not give uniform protection to the tank bottom, so some overlap must be included. In addition the anode should be extended to give reasonable protection to the side of the tank near its base.

d, h_2, L, R_2 and x in inches

$k_1 = 1/2.54$

r_h = resistance from anode to tank bottom

r_1 = resistance from anode to tank side

$R_2 = Lx/h_2$

cross sectional area at $x = \pi R_2^2$

$$r_h = k_1\rho \int_d^{h_2} \frac{dx}{\pi R_2^2} = k_1\rho \int_d^{h_2} \frac{dx}{\pi \left(\frac{L}{h}\right)^2 x^2} \qquad 7\text{-}1$$

$$= \frac{k_1\rho\,(h_2^2 - h_2d)}{\pi L^2 d} = \frac{0.1252\,\rho\,(h_2^2 - h_2d)}{L^2 d} \qquad 7\text{-}2$$

$r_1 = 0.00522\,\rho \log_e [2L(D-L)/dR]$ ohms per foot of rod (see eq. 6-13)

Assuming that r_h should not exceed 1.35 r_1, then

$r_h = 1.35\,r_1$ or $0.1252\,\rho\,(h_2^2 - h_2d)/(L^2d)$ 7-3

$$= 1.35 \times 0.00522\,\rho \log_e [2L(D-L)/dR], \qquad 7\text{-}4$$

$$h_2^2 - h_2d = 0.0563\,L^2d \log_e [2L(D-L)/dR] \qquad 7\text{-}5$$

$$h_2 = (d/2)\,(1 + \sqrt{1 + 0.2252\,(L^2/d) \log_e 2L(D-L)/dR}) \text{ inches} \qquad 7\text{-}6$$

Stub Anodes to Protect Tank Bottom

Area protected by outer ring of anodes

$$A_1 = \pi([R^2 - (R-2L)^2]) = \pi 4L(R-L) \qquad 8\text{-}1$$

Area protected by one anode

$$A_2 = \frac{A_1}{N} = \frac{\pi 4L(R-L)L}{\pi R} = \frac{4L^2(R-L)}{R} \qquad 8\text{-}2$$

Area remaining to be protected

$$A_3 = \pi(R-2L)^2 \qquad 8\text{-}3$$

Number of stub anodes required

$$N_s = A_3/A_2 = \pi R(R-2L)^2/4L^2(R-L) \qquad 8\text{-}4$$

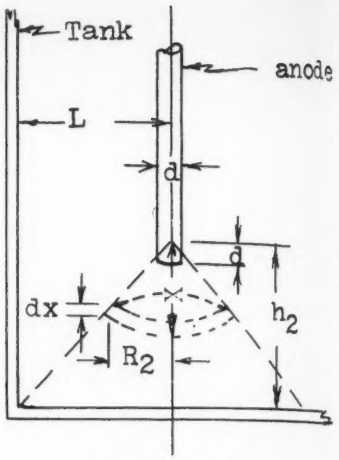

Figure for Appendix 2

Let $I_1 = \pi 2(R/12)\,i\,10^{-3}/(N)$ amperes per ft. of side anode 8-5

$$I_s = 2\pi \left(\frac{L}{12}\right)^2 i\,10^{-3} \text{ amperes for stub anode} \qquad 8\text{-}6$$

$N = \pi D_1/(D_1 - d_1) = \pi R/L$ number of side anodes 8-7

then $l_s = I_s/I_1 = \pi L/12 = 0.262L$ inches, length of stub anode 8-8

APPENDIX NO. 3

Anode Interference Factor

$\angle abo = 90°$

$$\alpha = \frac{360n}{2N} = \frac{180n}{N} \qquad 9\text{-}1$$

$$y = 2(R-L)\sin\alpha \qquad 9\text{-}2$$

$$\frac{X}{R-L} = \frac{2(h+L)}{X} \qquad 9\text{-}3$$

$$X^2 = 2(h+L)\,(R-L) \qquad 9\text{-}4$$

$$R^2 = X^2 + (R-L)^2 \qquad 9\text{-}5$$

$$= 2(h+L)\,(R-L) + R - L)^2 \qquad 9\text{-}6$$

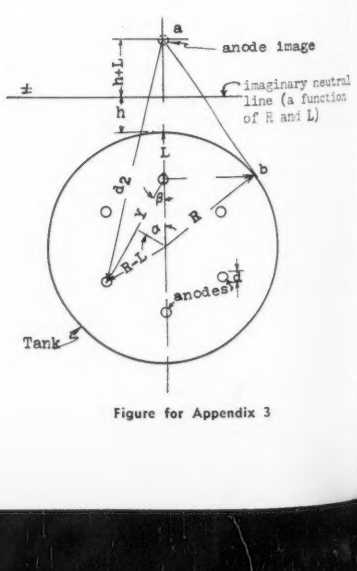

Figure for Appendix 3

$$h = L^2/2(R-L) \qquad 9\text{-}7$$

$$h+L = \frac{L^2+2L(R-L)}{2(R-L)} = \frac{L(D-L)}{2(R-L)} \qquad 9\text{-}8$$

$$\frac{2h+L}{L} = \frac{R}{R-L} \qquad 9\text{-}9$$

Resistance per foot of anode, to imaginary neutral line

$$r_2 = K \log_e \frac{4(h+L)}{d} = K \log_e \frac{2L(D-L)}{d(R-L)}$$

anode to ± 9-10

$$r_1 = K \log_e \frac{2h+L}{L} = K \log_e \frac{R}{R-L}$$

tank to ± 9-11

$$r_t = r_2 - r_3 = K \log_e \frac{2L(D-L)}{dR} = KG$$

anode to tank 9-12

$$G = \log_e \frac{2L(D-L)}{dR}$$ Geometric factor, for anode to tank resistance 9-13

K = previously defined

N = previously defined

n = number of spaces between anode No. 1 and anode being considered.

The effect of one anode on another is a function of the isopotential at anode No. 1, the anode under consideration, resulting from the voltage drop due to the current from other anodes flowing past anode No. 1. Assuming that the current "I" is equal to one ampere, then the isopotential e_n will be equal to "r," the resistance from the isopotential ring to the tank; that is,

$$e_n = r = K\,[\log_e d_2/y - \log_e R/(R-L)] \qquad 10\text{-}1$$

$$= K \log_e [d_2(R-L)/yR] \qquad 10\text{-}2$$

$$= Kg \qquad 10\text{-}3$$

where

$$d_2 = \sqrt{4[(h+L)^2 + y(h+L)\cos B] + y^2} \qquad 10\text{-}4$$

$$d_1 = \sqrt{D^2(\sin\alpha)^2 + [L(D-L)/(R-L)]^2} \qquad 10\text{-}5$$

Therefore, the geometric factor "g" for the isopotential reduces to

$$g = \log_e \sqrt{[L(D-L)/D(R-L)\sin\alpha]^2 + 1} \qquad 10\text{-}6$$

After calculating all the isopotentials that affect the anode under consideration, then the total potential at that anode is the sum of all the isopotentials superimposed on the equivalent isolated voltage of that anode; that is,

$$E = KG = K[G_1 + (g_2 + \ldots + g_n)]$$

The equivalent isolated voltage, therefore is $E_1 = KG_1 = K\,[G - (g_2 + \ldots + g_n)]$ 10-7

If E is the applied voltage, when all anodes are connected in parallel, then E_1 is that voltage which if applied to each anode alone would produce the isopotentials that, superimposed on E_1 will result in the applied voltage E. But, E_1 applied to each anode alone would cause the current I_1 amperes to flow from each anode. This current is less than I the current that would have resulted if E had been applied to each anode separately. The ratio of these two values is the interference factor F; that is,

$$F = I_1/I = E_1/E = K\,[G - (g_2 + \ldots + g_n)]/KG \qquad 10\text{-}8$$

$$= [G - (g_2 + \ldots + g_n]/G \qquad 10\text{-}9$$

From the above equation it is evident that the interference factor "F" is dependent on the geometric arrangement of the anodes and independent of the liquid resistivity, the applied voltage or the current value.

If the anode is not a continuous rod, but is made up of a string of shorter, equally spaced, sections of anode material coupled together to get the desired length, then the anode resistance becomes $r = r_1 k_2$ ohms. 10-10

where

$$k_2 = (l_2 + S_1)/(2l_2) \qquad 10\text{-}11$$

l_2 = length of each section of anode material; inches

S = center to center spacing of the sections of anode material; inches

APPENDIX NO. 4

Circuit Resistance

The total circuit resistance per foot of anode consists of, r_1 the resistance through the liquid, r_c the metal surface to liquid contact resistance, and r_a the resistance through the deposit on the anode and tank surfaces.

If the anode is of a metal, the sacrificed portion of which adheres to its surface, then the resistance through this coating must be included in the circuit resistance.

For Aluminum Anodes

$$d_a = \sqrt{d^2 - 0.00125\, I_h/\xi}$$ inches.
Diameter over the remaining metal. 11-1

I_h = the number of ampere-hours of service per ft. of anode.

ξ = efficiency of anode metal. Which in part depends on the purity of the anode metal. ξ varies from 0.13 to 0.6. For design purposes use 0.3.

$$d_b = \sqrt{(0.0065\, I_h/\xi) + d_a^2}$$ inches.
Diameter over $Al(OH)_3$ formed on surface of the anode. 11-2

The resistance of the aluminum coating

$$r_a = 0.00522\, \rho_a \log_e \frac{d_b}{d_a} \qquad 11\text{-}3$$

$$= 0.00522 \times 47{,}000 \log_e \frac{d_b}{d_a} = 245 \log_e \frac{d_a}{d_b} \qquad 11\text{-}4$$

ρ_a in Ω/cm^3 for $Al(OH)_3$, based on test data, varied from 38,000 to 58,000 with most values around 47,000, the value recommended for design purpose.

No resistivity values have been determined for the coating on the tank surface.

Metal surface to liquid contact resistance.

$$r_c = 0.00411\ B/d,\ \Omega/\text{ft. of anode.} \qquad 11\text{-}5$$

From test data the surface contact resistivity B appears to be $0.1 \times \rho$ and is so recommended. ρ = liquid resistivity.

The contact resistance at the tank surface generally is negligible.

For r_1 see equation 8-12.

$$r = r_1 + r_a + r_c \text{ ohms per ft. of anode} \qquad 11\text{-}6$$

r_t = combined resistance of all anodes per ft. of tank cross section, operating in parallel.

APPENDIX NO. 5

Rectifier Size

Rectifier current:

$$I_t = i\,[\pi D_1 h_1 + D_1^2/4) + A]\,10^{-3} \qquad 12\text{-}1$$

$$= i\,[\pi D_1 (h_1 + D_1/4) + A]\,10^{-3} \qquad 12\text{-}2$$

D_1 = inside tank diameter, in feet.

h_1 = height of over flow from tank bottom, in feet.

A = area of all other submerged surfaces such as braces, ladders, overflow pipes, etc., in square feet.

Rectifier voltage:

$$E = E_c + [(\pi D_1 i r_t)/F]\,10^{-3}, \text{ volts} \qquad 12\text{-}3$$

If the resistance of all anodes are equal then

$$E = E_c + [(\pi D_1 i r)/(NF)]\,10^{-3}, \text{ volts} \qquad 12\text{-}4$$

E_c = anode to tank open circuit voltage, which depends on the nature of the two metals, the magnitude of the current and the length of time the system has been in operation. If no information is available use $E_c = 0.5$ volts.

In selecting the voltage steps of the rectifier the finish, on the surface to be protected, must be taken into consideration. If the surface is painted then the voltage adjustments must permit keeping the drop across the paint film to one volt or less.

The voltage drop from the anode to the tank surface can be calculated, by substituting the value of i, based on equation 11-2, into equation 11-3 or 11-4. The difference between the calculated voltage and the measured voltage is taken as the voltage drop at the tank surface or the drop across the paint film.

NOMENCLATURE

α = 0.5 times angle between any two anodes under consideration
A = Area of submerged surface other than tank, sq. ft.
A_1 = Area of tank bottom protected by outer ring of anodes, sq. ft.
A_2 = Area of tank bottom protected by one anode, sq. ft.
A_3 = Area of tank bottom to be protected by stub anodes, sq. ft.
d = diameter of anode, inches
d_1 = diameter of ring of suspended anodes, ft.
d_2 = distance from image of reference anode to anode on isopotential to be calculated.
d_a = diameter of anode based on permissible resistance, inches
d_b = diameter of anode based on material consumed per year, inches
D = inside diameter of tank, inches
D_1 = inside diameter of tank, ft.
e = base of Naperian logarithm
e_n = isopotential or voltage drop from reference anode to some point in the solution.
E = applied voltage or operating voltage of rectifier.
E_1 = a given isopotential
E_c = anode to tank open circuit voltage
F = anode interference factor
G = geometric factor for reference anode
g = geometric factor for isopotentials for other anodes passing through reference anode
h = distance from tank to imaginary neutral line, inches
h_1 = height of overflow above tank bottom or maximum depth of liquid, ft.
h_2 = anode height above tank bottom, inches
i = current required per square foot of tank surface, miliamperes
I = current from any one anode to tank if all other anodes are disconnected and a voltage E is applied, in amperes per ft.
I_o = current required on tank side per foot height of tank, amperes
I_s = current required per stub anode, amperes
I_t = rectifier current rating in amperes
I_1 = current delivered per ft. of anode if in parallel with others and connected to the same voltage given for I above
k_1 = a constant, converting centimeters to inches
k_2 = ratio of resistance of a string of anodes as compared to a solid anode of equal over-all length
K = a constant for any one value of ρ. $K = 0.00522\,\rho$.
l_s = length of stub anode, inches
L = distance from anode to nearest tank surface, inches
N = number of uniformly spaced anodes in outer ring
N_s = number of stub anodes
n = number of spaces between reference anode and anode being considered.
r = combined resistance from anode to tank per ft. height of tank, ohms
r_1 = resistance through liquid from one anode to tank, ohms per ft. of anode
r_a = resistance through deposition anode, ohms per ft. of anode
r_c = resistance of metal to liquid contact, ohms per ft. of anode
r_t = resistance, all anodes in parallel, per ft. height of tank, ohms
r_2 = resistance through liquid from anode to imaginary neutral line, ohms
r_3 = resistance through liquid from tank to imaginary neutral line, ohms
r_h = resistance through liquid from anode to tank bottom, ohms
S = center to center spacing of the sections of anode material, inches
y = center to center distance between two anodes, or anode strings, under consideration, inches
ρ = electrical resistivity of the liquid in ohms per square centimeter per centimeter, Ω/cm^3
ρ_a = electrical resistivity of $Al(OH)_3$, Ω/cm^3

Any discussion of this article not published above will appear in December, 1960 issue.

Technical Papers on Corrosion Welcomed

Authors of technical papers on corrosion are invited to submit them for review without invitation to the Editor of Corrosion. Write for "Guide for the Preparation and Presentation of Papers" sent free on request to prospective authors.

High Temperature Lubrication In Reactive Atmospheres*

By R. L. JOHNSON, M. A. SWIKERT and D. H. BUCKLEY

Abstract

High temperature lubrication is usually dependent on direct chemical reactions involving the lubricated material, the lubricant, and the atmosphere. Extreme conditions of operation promote excessive chemical reactions that are manifested in corrosive wear. Friction and wear experiments show the influence of various atmospheres, lubricants, and materials, at temperatures to 1500 F. Gaseous halogen-substituted methane derivatives offer promise for solution of high temperature lubrication problems.

Data reported include effect of sliding velocity on friction of steel surfaces with and without performed films of Fe_3O_4, effect of air on lubrication of cast Inconel riders sliding against an Inconel disk with graphite as the lubricant, effect of number of chlorine atoms substituted in CF_4 on reactive gas lubrication of mild steel specimens, and effect of temperature on wear and friction of metals. 3.5.9

Introduction

LUBRICATION PROBLEMS at extremely high temperature levels are becoming increasingly common. In aircraft and missile propulsion systems, as well as other mechanisms, improved performance is usually accompanied by increased temperature levels for lubrication problems. Temperatures above 500 F are frequently encountered by components of lubrication systems in present turbojet engines. These temperature levels increase rapidly with greater supersonic flight velocities as well as greater powerplant output. Hypersonic flight vehicles operating in earth's atmosphere may encounter stagnation temperatures alone that are sufficiently high to destroy the usefulness of conventional structural metals. Thus, for lubrication systems, temperatures of 500, 1000, 2000 F and higher become important.

One of the primary effects of elevated temperatures on metals is increased chemical activity. In lubrication problems, chemical activity is always important. It can, however, be mainfested in corrosive or chemical wear and thus limit the usefulness of a machine element. At all temperatures, the oxidation of metals to form passive films or surface scale contributes directly or indirectly to most lubrication processes. These observations point up the fact that the atmosphere, the lubricant, and the lubricated material are interacting and basic components in lubrication problems at extreme temperatures. Corrosion technology is frequently applicable to the study of lubrication.

The objective of this paper is to present friction and wear data to emphasize the importance and behavior of interface films in lubrication with reactive atmospheres at extreme temperatures. Data are presented for temperatures to 1500 F with various materials, atmospheres, and lubricants. These data were obtained in the course of a number of experimental investigations conducted at the National Aeronautics and Space Administration's Lewis Research Center.

Fundamental Concepts

Several basic friction and wear concepts that have been accepted by many investigators are important to understanding phenomena occurring in lubrication with surfaces in solid contact (boundary lubrication). These concepts are based on a microscopic consideration of surface character and usually are essentially independent of the gross geometry of the mechanical parts.

A primary concept is that contact of surfaces occurs between asperities or protuberances. All surfaces have irregularities or roughness; however, the common measures of roughness may define only whether a surface is rough on a macroscopic, a microscopic, or an atomic scale. It follows that the real area of contact is a minute fraction of the apparent area in contact (the apparent area of contact is defined by the gross geometry of the mating parts). As surfaces come into solid contact under light load, very few of the asperities or high points may actually touch. With increasing loads, the initial points of contact deform elastically, then plastically, and additional asperities assume a portion of the total load. The local stress of the initial asperities in contact is then at the elastic limit (or yield stress in compression) of the weaker of the materials in contact. Thus, the maximum real surface stress will usually be the yield stress, and the increased total load simply increases the real area of contact.

The common laws of friction (Amonton's Laws) are usually valid because plastic deformation determines the real area in contact. They are not valid when deformation is primarily elastic or when the load, materials, and geometry are such that the real area and the apparent area in contact are equal.

The most widely accepted expression for these concepts has been advanced by Bowden and Tabor[2]. This expression shows that friction coefficient (f), which is defined as the ratio of friction force (F) to normal load (W), is equal to the shear strength (s) of the softer metal in contact divided by the yield strength (P) in compression of the weaker metal. Where A is the real area of contact, these simple concepts may be related as follows:

$$f = \frac{F}{W} = \frac{As}{W} = \frac{As}{AP} = \frac{s}{P}$$

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Thin surface films, such as lubricants or other deposited or reactive coatings, reduce friction by lowering effective shear strength. The important bulk yield strength factor, which determines shear area, is a function only of the base metal substrate so long as the film is very thin. Among the most common surface films important to lubrication are the surface oxides. The type, thickness, and character of such films are a function of thermal history, chemical kinetics, and thermodynamics of the system; surface temperature is especially important.

The energy consumed in friction during sliding of solid surfaces must be dissipated as frictional heat. With the real area of contact a small fraction of the total surface area, the heat flux at the surface asperities may be extremely high. It has been demonstrated[2] that surface temperatures of 500 to 1000 C (932 to

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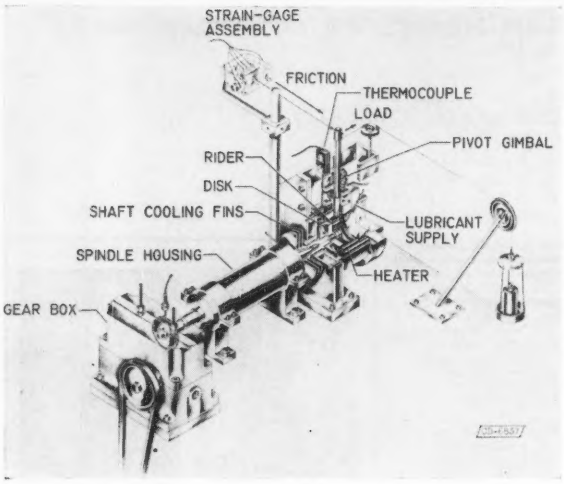

Figure 1—High temperature friction apparatus.

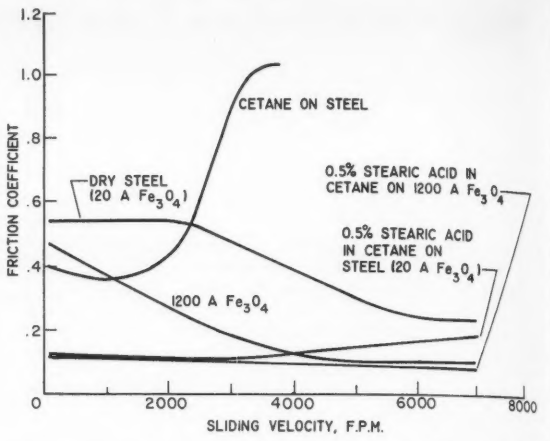

Figure 2—Effect of sliding velocity on friction of steel surfaces with and without preformed films of Fe_3O_4 1200 Angstrom units thick. No lubrication, purified cetane and 0.5 percent stearic acid in cetane. Load, 269 grams; ⅛-inch-radius rider on flat disk, 75 F.

1832 F) above the ambient temperature can easily be experienced. With chemical activity a function of temperature, it is apparent that surface reactions will preferentially occur at the local high temperature areas on the surface asperities. Excessive reactions give chemical attrition or corrosive wear, so that an optimum chemical activity of the surface must be sought to obtain effective surface protection from both adhesive and corrosive wear.

Apparatus

The experimental data to be presented were obtained with several devices for studying friction and wear over a wide range of conditions. Basically, these devices all employed loaded and elastically restrained hemispherical rider specimens sliding in a circumferential track on the flat surface or rotating discs. A typical apparatus, which was used to obtain much of the data presented, is shown in Figure 1. The 2½-inch-diameter rotating disk specimen was surrounded by a housing containing cartridge heaters capable of providing specimen temperatures over 1500 F. Specimens were contained within a sealed chamber that allowed control of atmosphere. Surface speeds up to 10,000 fpm could be obtained. The usual rider specimen had a 3/16-inch radius contacting the flat surface of the disk specimen. With the loads and metals used, the initial Hertz surface stresses were from about 50,000 to 160,000 psi.

Results and Discussion

The normal oxide films present on metallic surfaces, such as steel, are of vital importance in boundary lubrication with conventional liquid lubricants. The data of Figure 2 show the effect of sliding velocity on friction coefficient for steel specimens with varied conditions of lubrication. The surface described as dry steel had an initial surface oxide film 20 Angstrom units thick that was essentially Fe_3O_4; with sliding velocities greater than 2000 fpm, surface temperatures were sufficient to promote the formation of stable FeO. This was one of the factors that caused the substantial reduction in friction between 2000 and 6000 fpm.[2] Unlubricated sliding steel specimens with a performed film of Fe_3O_4 1200 Angstrom units thick showed substantially lower friction than the normal dry steel.[4]

In experiments with highly purified cetane on steel (Figure 2), the cetane served to mask the continued formation of surface oxide films.[5] Thus, when the original oxide layers were destroyed by operation at increased sliding velocities, the friction was more than doubled.

Stearic acid may be considered typical of many of the polar molecules present in liquid lubricants. Experiments with 0.5 percent stearic acid in cetane on normal steel and on steel with a Fe_3O_4 performed film 1200 Angstrom units thick are also presented in Figure 2. It is quite apparent, at sliding velocities above 3000 fpm, that the presence of the thicker oxide film is beneficial in gaining more effective lubrication. These data are discussed in more detail in reference 6.

The data of Figure 3 show the effect of atmosphere on lubrication with graphite as a solid lubricant at 1000 F. It is generally accepted that graphite lubricates at normal temperatures because of adsorbed moisture or vapor on the graphite particles. The data of Figure 3 show that, with Inconel specimens, graphite will not lubricate in a reducing atmosphere, but will lubricate in air, and that, at this temperature (1000 F), graphite is more effective in dry air than in moist air. The changes in atmosphere of this experiment were made to influence the tendency of oxide films to form on the metal surfaces. In this and other experiments,[7] it has been demonstrated that effective lubrication can be obtained with graphite at elevated temperatures if an oxide film of the proper type and thickness is obtained on the lubricated surface, regardless of the presence of moisture. The oxide film is believed to influence the adherence of graphite to the surface.

Conventional gear lubrication problems frequently require the use of reactive lubricant additives in liquid lubricants. Typical additives contain reactive atoms of sulfur, chlorine, or phosphorus. These additives function because the flash temperatures of surface asperities are sufficiently high to rupture the adsorbed additive molecule to release the active atoms and then to provide energy to aid reaction with the lubricated metal. The reaction products provide the actual lubrication. These products may be either low-shear-strength compounds or compounds that promote the formation of surface eutectics.

Wherever lubrication depends on direct chemical reaction, corrosive wear may be a problem. Data of reference 8 show that a significant level of activity was needed for lubrication at high surface speeds. The limiting sliding velocity for effective lubrication was directly related to known chemical activity of the additives.

Halogen-substituted methane and ethane derivatives are being considered as possible gaseous boundary lubricants for extreme temperatures.[9, 10] The lubrication mechanism for these materials is basically the same as for the reactive additive containing liquid lubricants previously mentioned. Data for fully halogenated methane derivatives presented in Figure 4 for steel surfaces at room temperature show that two chlorine atoms were necessary in fluorine- and chlorine-substituted methane to obtain minimum friction. In these[9, 10] and subsequent studies,[11, 12] no evidence has been found that the carbon-fluorine bond was ruptured.

Wear and friction data with dichlorodifluoromethane lubricating hardened tool steel at various temperature levels are presented in Figure 5. Extremely low friction at temperatures above 400 F suggests local surface melting of the reaction film ($FeCl_2$ plus 15 percent $FeCl_3$ per X-ray diffraction and chemical analyses of reference 10). The rapid increase in wear above 600 F was corrosive in nature

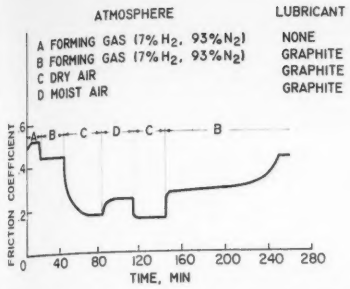

Figure 3—Effect of air or reducing atmosphere on lubrication of cast Inconel riders sliding against an Inconel X disk with graphite as the lubricant. Temperature, 1000 F; sliding velocity, 5.7 feet per minute; load, 40 pounds on three 3/16-inch-radius rider specimens.

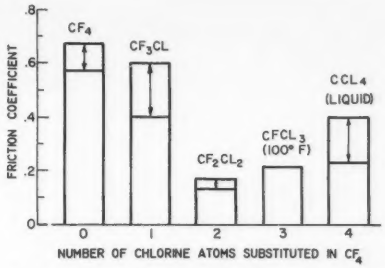

Figure 4—Effect of number of chlorine atoms substituted in CF_4 on reactive gas lubrication of mild-steel specimens. Temperature, 75 F; sliding velocity, 120 feet per minute; load, 1200 grams on a 3/16-inch-radius rider specimen.

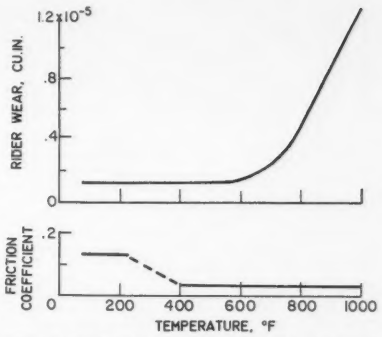

Figure 5—Effect of temperatures to 1000 F on wear and friction of hardened M-1 tool steel lubricated with dichlorodifluoromethane. Sliding velocity, 120 feet per minute; load, 1200 grams on a 3/16-inch-radius rider specimen; duration, 1 hour.

and resulted from excess chemical activity.

The data of Figure 6 were obtained with bromine-substituted fluorinated methanes. By using less-reactive high-temperature alloys and by using mixtures of gases, it is possible to obtain low wear at temperatures from 75 to 1200 F. Wear for the 1:1 mixture of CF_3Br and CF_2Br_2 was lower at all temperatures than for either gas alone. This result suggests a synergistic effect with the gas mixture.

A number of compensating factors can be utilized with reactive gas lubricants to obtain effective lubrication without corrosive wear. One factor is the type of reaction film formed. Ideally, the reaction film should be passive to further surface reaction by the lubricating gas; this situation is not generally achieved. The reaction film should have low shear strength; thus the frictional heat developed would be less, and the resulting lower surface temperature will reduce the rate of reaction. Low shear strength is usually obtained with crystalline solids having layer lattice structure.[13] Another factor of probable importance to surface reactivity is that the amount of gas adsorbed on the surface will decrease with increasing temperatures and thus compensate for the greater reactivity at higher temperatures. Rate of reaction can be controlled to some extent also by the use of catalyst additions such as the addition of SF_6 to CF_3Br indicated in Figure 6. Furthermore, alloy constituents can be added to the lubricated metals to control reactivity. An example might be the use of silicon additions to nickel reported in reference 14 to promote the formation of desirable surface films. Mechanical considerations which influence surface temperature phenomena can also be utilized to control surface reactions. For example, a surface in intermittent contact should be of more reactive material, to obtain effective lubrication, than a surface in continuous contact. Continuous contact might result in sustained high temperature levels from friction heat, and thus a reactive material might be subject to corrosive wear.

In most instances, the high temperature lubrication mechanisms with alloys are difficult to resolve. Many common alloys have chemical, physical, and mechanical properties at elevated temperatures that are inadequately understood. The chemical, physical, and mechanical properties of pure copper are as well understood as those for any other metal or alloy. A series of experiments was therefore performed at temperatures from 75 to 1500 F to relate the known properties of copper to its behavior as a slider material.

Figure 7 presents wear and friction data obtained with electrolytic copper (99+ percent) rider and disk specimens operated in air and in air containing 5 percent sulfur dioxide. These data were obtained with a 600-gram load, which, with the 3/16-inch-radius rider specimen, gives an initial Hertz surface stress of 91,000 psi at 75 F (58,000 psi at 1000 F and 51,000 psi at 1400 F). Preliminary wear data at room temperature showed that rider wear increased linearly with greater loads over the range from 150 to 1150 grams. This observation is consistent with the normal concepts of adhesive wear[1]

Substantial changes in wear and friction occurred with copper when runs were made in air at higher temperature levels (Figure 7). In the ambient temperature range from 75 to 475 F, wear increased sharply with temperature while friction remained high ($f = 1.4$ to 400 F). From 75 to 475 F, increased formation of Cu_2O films with high temperature was visibly apparent by interference color changes of surfaces. Above 475 F the formation of a double oxide film CuO on Cu_2O on copper was verified by X-ray diffraction. Both wear and friction decreased as the proportion of CuO in the oxide layers increased. Wear increased in a critical manner above 1400 F as the result of scaling and subsequent reoxidation of the copper. Thus it becomes apparent that CuO is the more desirable copper oxide for slider surfaces and that corrosive wear, under these conditions of operation, does not become a primary problem at temperatures to 1400 F.

To further emphasize the importance of oxidation reactions for copper, 5 percent sulfur dioxide was added to the air atmosphere. The SO_2 was considered to act as a catalyst to promote the oxidation reaction.[15] The wear results (Figure 7) suggest that the SO_2 did promote surface oxidation. The same characteristic rider wear curve was obtained as with air but with the initial wear maximum at 300 F rather than 475 F. X-ray diffraction analysis indicated that the surface film materials were essentially the same in air as in air plus SO_2. The primary effect of the SO_2 addition to the atmosphere was the formation of the double copper oxide at lower temperatures.

A characteristic relation between wear and friction that suggests corrosive wear was noted at 1500 F (Figure 7). In this instance, low friction resulted from complete separation of nascent surfaces by copious formation of the reaction film. Repeated scaling and rapid reoxidation contributed to the excessive corrosive wear; thus, it becomes apparent that physical as well as chemical factors are important. At 1500 F, corrosive wear was greater with SO_2 present. It is of interest, however, that the temperature for the incidence of excessive corrosive wear was similar (> 1400 F) for both air and air plus 5 percent SO_2.

Any questions as to the relative role of the reaction film and the substrate metal in wear and friction trends can be partly resolved by providing different reaction films on the same base metal. For example, the wear maximums around 400 F might be related either to metallurgical transformation or to the characteristics of the surface reaction film. Metallurgical transformations are likely because 400 F is the beginning of the temperature range for annealing of copper. Hot-hardness data[16] showing a linear reduction in Brinell number with increasing temperatures to almost 1000 F also indicate physical changes. With H_2S present in oxygen-deficient atmospheres such as 5 percent H_2S in dry nitrogen, the reaction film (identified by X-ray diffraction) on copper surfaces was sulfide over the entire temperature range to 1500 F. In air and in air plus SO_2 the reaction film was oxide.

The differences in wear and friction data in air and in the H_2S-N_2 mixture presented in Figure 8 are the results of the different surface reaction products. From Figure 8 it immediately becomes apparent that copper sulfides are more effective in reducing wear than the oxides.

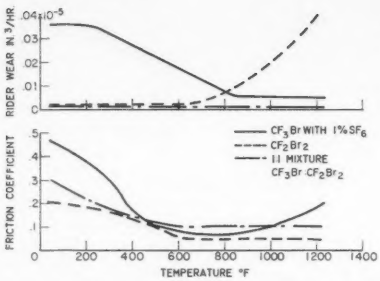

Figure 6—Effect of temperatures to 1200 F on wear and friction of a cobalt-base alloy (98M2 Stellite) and a nickel-base alloy (Hastelloy C) lubricated with CF_3Br, CF_2Br_2, and an equal mixture of the two gases. Sliding velocity, 120 feet per minute; load, 1200 grams on a 3/16-inch-radius rider specimen; duration, 1 hour.

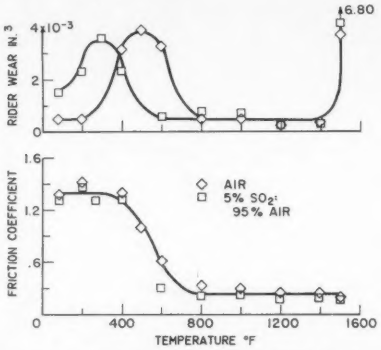

Figure 7—Effect of temperatures to 1500 F on wear and friction of electrolytic copper rider and disk specimens in air and in air containing 5 percent SO_2. Sliding velocity, 100 feet per minute; load, 1200 grams on 3/16-inch-radius rider specimen; duration, 30 minutes.

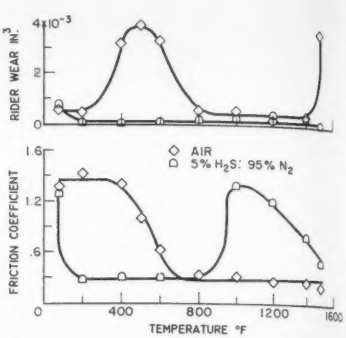

Figure 8—Effect of temperatures to 1500 F on wear and friction of electrolytic copper rider and disk specimens in air and in nitrogen containing 5 percent H_2S. Sliding velocity, 100 feet per minute; load, 1200 grams on 3/16-inch-radius rider specimen; duration, 30 minutes.

The oxides, however, have greater chemical stability than the sulfides.[17] In the lower temperature range (< 800 F), the sulfide ($\propto Cu_2S$) provided lower friction than the oxide. After runs at 1000 F and higher temperatures, the X-ray diffraction patterns of reaction products indicated distortion or possible phase transformation of the Cu_2S. The abrupt increase in friction was most probably the result of crystal transformation of the Cu_2S, which influenced shear strength. Substantial dissociation of H_2S begins at temperatures above 1000 F.[18] The subsequent decrease in friction and wear at those higher temperatures may be indicative of the increasing ease with which surface reactions occur in the presence of the partly dissociated H_2S. The relatively low wear experienced with the H_2S-nitrogen mixture is a further indication that compensating factors in surface reactions can prevent prohibitive corrosive wear.

These data with copper specimens (Figures 7 and 8) serve to illustrate the importance of surface reaction phenomena in friction, wear, and lubrication. Many of these observations are of importance in the area of corrosion as well. It becomes quite apparent that, at extreme temperatures, gaining effective lubrication can well be a matter of controlled corrosion.

Concluding Remarks

A review of some NASA lubrication studies and the introduction of specific research data provide the basis for the following generalizations on lubrication at elevated temperatures:

1. The formation of surface films in atmospheres with the potential for oxidation-type reactions is a basic component of lubrication mechanisms at elevated temperatures. Physical, chemical, and mechanical properties of the films and the substrate metals at various temperatures up to the maximum of 1500 F reported herein determined how friction, wear, and lubrication are influenced. Surface reactions are necessary for effective lubrication but at extreme temperatures can be excessive and result in corrosive wear.

2. An effective extreme-temperature lubrication method based on surface film formations from reactive gases can be obtained. For example, a reaction product with layer lattice crystal structure such as $CoBr_2$ or $NiBr_2$ has low shear strength and will usually give low friction.

Cobalt-base (e.g., 98M2 Stellite) and nickel-base (e.g., Hastelloy C) high-temperature alloys sliding in a lubricating atmosphere of bromine- and fluorine-substituted methane or ethane derivatives such as CF_3Br and CF_2Br_2, will form the low-shear-strength bromine compounds. Optimum reaction characteristics will provide low friction and wear, but the possible incidence of corrosive wear because of excessive reactivity remains a critical problem.

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DISCUSSION

Comments by Roy E. Dial, Grand Island, New York:

Have you considered "KT" SiC as a high temperature bearing material, dry or in lubricant? It has been tested as a dry bearing at 1800 F successfully "without galling." The material was used as ⅜ inch diamond-machined balls made to a sphericity of 0.0000050 inch. "KT" SiC has high thermal conductivity (5½ times Inconel) and high compressive strength (250,000 psi at room temp.) It also is very hard and abrasive resistant, and can be highly polished.

It is suggested that boron nitride powder be considered as a dry lubricant for high temperature.

Reply by Robert L. Johnson:

We have not obtained data with Carborundum's "KT" silicon carbide. Other forms of silicon carbide have been considered, however, and can have useful characteristics for bearings and seals. Primary limitations on use of any SiC are the problems of fabricating and handling such a hard and brittle material. Local chipping and fragmentation have been noted. Perhaps the "KT" silicon carbide may be less subject to these difficulties because it does have improved physical properties.

Our experience with boron nitride powder as a solid lubricant indicates that its ability to lubricate sliding metals is limited. Some reduction in friction was obtained above 1350 F where oxidation of the BN was noted; lubrication may have been provided by the oxide alone or by the oxide serving as an adjunct to promote adhesion of the BN to the metal surfaces.

Development of Thin Metal Film Corrosion Indicators*

By DAVID ROLLER and G. H. ROHRBACK

Abstract

An account is given of the development of visual and electrical corrosion indicators which utilize thin metal films vacuum-deposited on non-metallic substrates. Data are presented showing the behavior of these indicators in corrosive atmospheres. Although the indicators were developed primarily to detect and mark corrosive conditions in packages and storage areas, other possible applications are described briefly. Data reported include comparative corrosion information on electrical cards and buttons in various humid atmospheres, condition of visual and resistance thin film indicators at first sign of corrosion on hardware items, and behavior of polyvinyl resistance card indicators at different sensitizations at 25 C and 60 percent relative humidity.

The paper presents the progress of this development program from the feasibility studies through the final development of manufacturing protypes. 3.2.3

Roller

Rohrback

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GILSON H. ROHRBACK has been associated with Magna Products, Inc. since 1952. Since 1954 he has been President of Magna Products, Inc. and Manager of its Research & Development Division. He was previously associated with the California Research Corporation. He has a Ph.D. in chemistry from the University of Washington.

Introduction

THE DEVELOPMENT of thin metal film corrosion indicators is an outgrowth of preliminary investigations carried out by the Air Force at the Wright Air Development Center, Dayton, Ohio. The utilization of vacuum deposited iron films on glass and plastic substrates was investigated as indicators of humid environments. This work[1] showed that vacuum deposited thin iron films will corrode in humid environments at rates which were apparently faster than gross metallic hardware. During the corrosion process the iron films changed both their visual and electrical properties. These changes were eminently apparent even to untrained observers. The work concluded that sensitive, easily used, and inexpensive corrosion indicators could be developed for packaging and storage applications using thin metal films as the sensing elements.

A thin metal film offers two unique advantages when considered as a corrosion indicator. The surface of steel shim stock can be made as sensitive to a corrosive environment as the surface of a vacuum deposited metal film which is only one thousand angstroms ($1000\text{Å} = (3.937 \times 10^{-6}$ inches$) = .003937$ mils or $= .0001$ mm) thick.

Either shim stock or a metal film could be used as a corrosion indicator. An advantage of a thin metal film is that the electrical properties change proportionately much more than does bulk metal during the corrosion process. Thus, simple instrumentation can be devised to monitor the change in electrical resistance (for example) of a thin metal film. Another advantage of a metal film is that during the corrosion process the thin film changes its appearance and color drastically, and the metal film itself is disrupted. Thus when the metal film is deposited on a transparent substrate, these changes to the metal film can be easily observed either by looking directly at the metal film or by looking at the film through the transparent substrate. These two advantages might be effectively used in properly designed corrosion indicators.

The Air Force Wright Air Development Center subsequently initiated a research program at the Engineering Experiment Station, Georgia Institute of Technology, to study the effect of exposing various thin metal films to various atmospheres. In addition, basic studies of those parameters affecting thin metal film corrosion rates were undertaken. This effort has culminated in two excellent reports[2,3] regarding the behavior of many types of thin metal films in corrosive environments. Both single and multimetal films have been evaluated under a variety of conditions. In addition, techniques of metal film deposition, aging treatments, substrates, packaging, and a variety of other factors have been investigated. Work at Georgia Tech provided a basis for further work, particularly design and manufacturing studies of actual corrosion indicator hardware.

The Air Force Air Material Command Aeronautical Systems Center initiated a program in March 1958 to undertake manufacturing studies of both visual and electrical thin metal film corrosion indicators.[4] The indicators to be developed were designed to: (a) Determine the corrosivity of the environment; (b) Integrate the information over time; (c) Be more sensitive to a corrosive environment than the packaged hardware item; and, (d) Be able to forewarn operating personnel as to the corrosivity of the environment so that necessary corrective action could be taken before loss by deterioration of a packaged item occurred. The effort was to develop suitable thin film indicators and to develop inexpensive manufacturing methods to produce them in sufficient quantity for field evaluation in packaging and storage application.

Several other investigations have been made on the application of thin metal film corrosion indicators. The Taft Sanitary Engineering Center has been utilizing corrosion indicators to detect and measure atmospheric pollutants. Vacuum deposited metal films of iron, copper, aluminum and silver on glass substrates have been exposed in a number of locations. Each metal film specimen was analyzed both visually and electrically. Corrosion rates as measured by these indicators apparently correlated with other techniques of measuring air pollution.[5]

Certain preliminary tests at Magna Products, Inc. and at Melpar Inc. of Virginia have shown other potential uses for thin metal film corrosion indicators. Melpar has utilized thin metal film indicators to detect corrosive environments within their laboratories and manufacturing areas. The response of electrical equipment was being affected by such environments. The erratic response was traced back to corrosive action which occurred during manufacture. The indicators have been used to forewarn personnel of the existance of corrosive conditions within the manufacturing area.[6] From preliminary studies it has been shown that thin metallic films will corrode in a simulated jet fuel (kerosene) which has been contaminated with very small percentages of water. The indicators do not corrode in anhydrous kerosene. In other tests various types of volatile corrosion inhibitors (VCI) have been tested utilizing thin film indicators.

The time needed for various VCI materials to protect iron films from corrosion have been briefly examined. Some work has been done on the time required for corrosion to start when the VCI material is removed. In addition preliminary work has shown that it might be possible to develop a thin metal film indicator which will react specifically with very low concentrations of ozone in air.

Test Program

An appropriate sensing element has been shown to consist basically of a thin metal film deposited over a suitable substrate. Thin metal films of iron were found to be most suitable as the sensing element in environments which are considered to be low in corrosive potential such as is to be found in normal packaging and storage application. Corrosion rates of films of nine metals exposed to air atmospheres of controlled temperature and humidity have been studied.[2]

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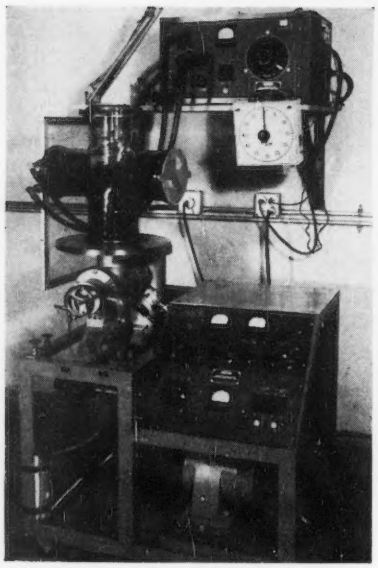

Figure 1—Vacuum deposition system.

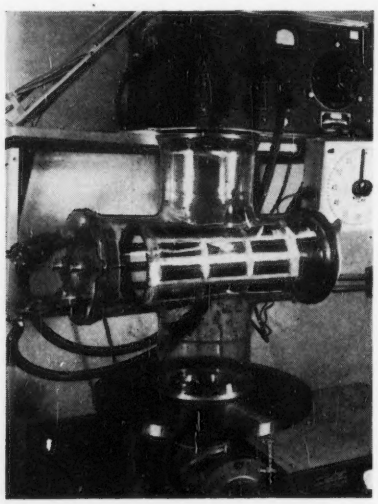

Figure 2—Vacuum deposition chamber.

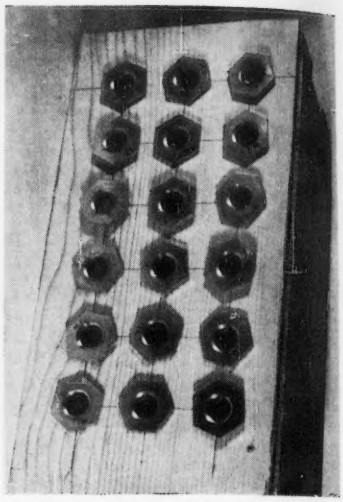

Figure 3—Stagnant air chamber open on one side showing mounted window indicators prior to exposure in test environment.

Iron and manganese films were considered to be most useful as corrosion indicators, while other metals studied were found to corrode too quickly (calcium, strontium); too slowly (copper, nickel, cobalt); or too irregularly under the controls available (zinc, magnesium). Iron was selected as being the most ideal indicator sensing element metal, particularly for packaging and storage applications. Furthermore, an iron film would have corrosion characteristics similar to those of steel.

In producing the corrosion indicator, the sensor film of iron has been deposited on selected substrates using conventional vacuum deposition techniques.[7] The vacuum equipment used in this program consisted of a Kinney PW-400 vacuum pumping system and a two KVA packaged power supply for the low voltage evaporating power source. A mechanical pump and 4" fractionating diffusion pump were used to obtain high vacuum in a short period of time. Standard accessory equipment was used, including a liquid nitrogen trap, thermocouple gauges, and an ionization gauge. Figure 1 is a photograph of the vacuum deposition system. Figure 2 is a photograph of the vacuum deposition chamber. The vacuum deposition method, which proved most satisfactory, utilized the following procedure:

a. Prepare substrates by desired cleaning method.

b. Mount substrates in mask.

c. Insert mask in vacuum chamber.

d. Wind predetermined quantity of material to be evaporated on tungsten filament.

e. Insert filament holder in vacuum chamber.

f. Evacuate chamber with mechanical roughing pump to approximately 100 microns vacuum.

g. Connect oil diffusion pump to chamber and evacuate to desired pressure, which was approximately 5×10^{-5} mm. Hg.

h. Connect power supply cables to external filament terminals and apply voltage required to vaporize iron on filament. (5-30 seconds time required, depending on film thickness desired.)

i. Decrease voltage applied to filament and continue heating substrate and deposited film if post-deposition vacuum heat treatment is desired. This usually results in a more adherent film.

j. Vent chamber and remove mask and substrates.

Both visual and electrical prototype corrosion indicators were tested in a variety of environments. Humidity, temperature, various types of vapor corrosion inhibitors, acid vapors, and other parameters were varied. Several environmental test chambers, many large glass jars, and actual packages were used in the test program. Desired environments were produced using either standard humidity chambers or chemical solutions. Several thousand iron films in many configurations have been tested to date. Qualitative tests were made comparing the corrosion rates of bulk metal hardware with prototype corrosion indicators. Two different metal items were used: (a) a raceway and ball bearings; and, (b) highly polished shim stock test coupons.

The degree of corrosive attack was determined either visually or electrically, and sometimes both ways simultaneously, depending on the type of specimen being used. Many tests have been made in which both visual and electrical indicators were exposed simultaneously to the same environment. An observer's visual estimates of the percent of the area corroded varied by 10 to 20 percent, though any one observer was usually quite consistent. Electrical readings were made with an electrical resistance measuring device.[8] This instrument uses a modified Wheatsone bridge to measure small changes in the metal film's electrical resistance.

As many as six replicate specimens were tested at one time to determine indicator accuracy and precision. Periods of time required for initial and moderate corrosion to occur were reproducible within about 20 percent. Under certain conditions, the scatter of data was greater or less than this. For example, specimens exposed to highly corrosive conditions would show less apparent scatter than replicate specimens exposed to mildly corrosive conditions. The spread in experimental data can be attributed to many factors, particularly the randomness of the corrosion process, indicator manufacturing variabilities, and experimental difficulties, such as observer bias.

Some interesting observations were made during the evaluation of the indicators. The following comments are considered pertinent to the test program:

a. Sulfuric acid solutions are generally satisfactory[9] for control of relative humidity, but were discarded because of the possibility of corrosive, volatile acid gases. A volatile corrosion inhibitor (VPI, Shell Development Company) and, occasionally, traces of sensitizing salts reacted with the sulfuric acid solution.

b. Glycerol solutions[10] were tried but the humid environment produced was too sensitive to temperature changes. The temperature coefficient was of the order of 1 percent relative humidity per degree F, in contrast to that of the sulfuric acid solutions, which was about 0.2 percent relative humidity per degree F.

c. Solutions of potassium hydroxide[11] were tried and found to be free of all objections. Almost all tests in jars were carried out over KOH solutions.

d. Air flow rates were found to have a marked effect on indicator corrosion rates. The faster the air moved the greater the corrosion rate. Stagnant air chambers were used in all tests to eliminate this variable. Stagnant air environments also most closely simulate conditions to be found in packaging and storage conditions. Therefore, when in-

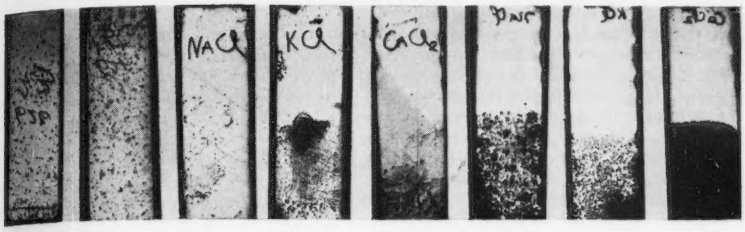

Figure 4—Effect of various salt sensitizations on iron film corrosion. Description of samples is as follows: (1.) Mylar Substrate half dipped in 0.25 percent $CaCl_2$ in acetone (48 hours of exposure at 100 F—70 percent relative humidity); (2.) Mylar Substrate half dipped in 0.25 percent KCl in alcohol (48 hours of exposure at 100 F—70 percent relative humidity); (3.) Mylar Substrate half dipped in 0.25 percent NaCl in acetone (48 hours of exposure at 100 F—70 percent relative humidity); (4.) Mylar Substrate half dipped in 0.025 percent $CaCl_2$ in acetone (96 hours of exposure at 70 F—40 percent relative humidity); (5.) Mylar Substrate half dipped in 0.01 percent KCl in alcohol (90 hours of exposure at 70 F—40 percent relative humidity); (6.) Mylar Substrate half dipped in 0.025 percent NaCl in acetone (96 hours of exposure at 70 F—40 percent relative humidity); (7.) Glass Substrate—no cleaning (144 hours of exposure at 70 F—40 percent relative humidity); (8.) Glass Substrate cleaning by passing slide through Bunsen flame only (144 hours of exposure at 70 F—40 percent relative humidity.

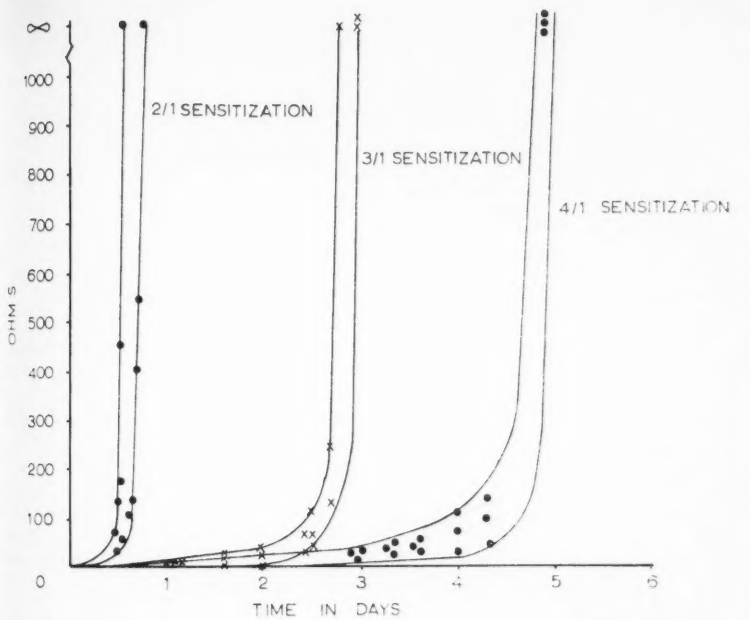

Figure 5—Behavior of polyvinyl resistance card indicators at different sensitizations at 25 C and 60 percent relative humidity.

dicators were tested in humidity cabinets, they were first placed in jars and boxes with loose fitting covers as shown in Figure 3.

Test Results and Discussion

Replicate pure iron films were found to corrode erratically in humid environments. Rusting of the pure iron films was greatly affected by many variables, particularly film contamination. Other investigators[1, 2, 3, 12] showed that pure iron films which were completely uncontaminated, did not corrode in normally "corrosive" humid environments. Contamination usually occurred in a random fashion resulting in erratic response. It seemed that a contaminated metal film which corroded at approximately the same rate as inadvertently contaminated bulk metal was desirable as the sensing element. This suggested that the sensing element of the corrosion indicator should first be artificially contaminated.

The use of an artificially contaminated film which would corrode uniformly is not entirely new. Certain specification tests for preservative oils and greases utilize a polished metal plate of mild steel. A "synthetic fingerprint" of salt is placed on the metal plate, which is then covered with the oil. The purpose of the test is to be sure that the preservative oil or grease can protect the steel plate and contaminated surface from corrosion when placed in a humid test environment. Likewise, a corrosion indicator for packaging really should denote the possibility of corrosion to an inadvertently contaminated surface. If the packaged part is not contaminated it will not corrode and no indicator would be needed. It is for the contaminated surface, which can corrode, that an indicator is most desired. By this reasoning an artifically contaminated or sensitized metal film indicator was desired.

Many salts were investigated to determine their effect on deposited iron films. (See Figure 4.) Sulfate and nitrate salts produced indicators with longer response times, but ones which did not corrode completely, even after several months. Nitrite and chromate salts, which are known corrosion inhibitors, produced indicators which did not corrode. Either erratic results or spotty corrosion products occurred when halide or metal ions were substituted for the ammonium ion. Additional work in this area has been undertaken by Georgia Tech.[3]

Ammonium chloride was found to be most satisfactory in producing a uniform and reproducible pattern of corrosion. The rate of corrosion was fairly fast, however. To slow down the initial rate of corrosion, desirable during manufacturing and packaging, it was found that the addition of sodium chromate was effective in inhibiting the initial corrosion. Various combinations and methods of sensitizing iron films with ammonium chloride and sodium chromate were undertaken.

A sensitized film of about 4 parts sodium chromate to about 1 part ammonium chloride, deposited onto the vacuum deposited film, furnished a uniform and reproducible pattern of corrosion. Extensive testing of this combination has been undertaken. At relative humidities below about 40 percent at 25 C, no corrosion occurs. At higher humidities and temperatures, the corrosion rate increases. Sensitizing with proportionately greater amounts of ammonium chloride decreases the time for corrosion to occur on the metal film as shown in Figure 5. The sodium chromate acts as an inhibitor of corrosion and produces acceptable delay periods.

The effect of variations in the amount of salt deposited for sensitization has been briefly investigated. Large amounts which are plainly visible cause the film to begin to corrode rapidly within 24 hours at any relative humidity above 35-40 percent. As the amount of salt is decreased, a point is reached where a higher relative humidity is required before rapid corrosion will take place within 24 hours. A gradation of reaction rates is found on films bearing non-uniform salt deposits. Corrosion begins first where salt is heaviest and spreads over a period of time to areas with lower salt concentrations. For indicators to perform reproducibly, control of this factor is essential during manufacture. Good results have been obtained with 4×10^{-5} grams of ammonium chloride per square inch of metal film surface area. A 50 percent variation around this figure is not critical. Smaller amounts produce spotty corrosion and larger amounts further reduce response time.

Other factors which related to the design of metal film corrosion indicator were also investigated. Composition of the iron film is a critical factor for unsensitized metal films. Small impurities

and discontinuities form electrochemical cells randomly across the metal film and these are difficult, if not impossible, to control. By sensitizing the iron film utilizing suitable salts, the purity of the iron and subsequent composition of the metal film is not an important factor.

Variations in size and shape of the areas covered with the sensitized metal film were studied, and little effect was observed. Bi-metallic configurations, such as copper overlaid with iron, were not studied under this program. However, considerable work on bi-metallic films has been undertaken.[2,3] Generally the rate of corrosion is increased due to the initiation of galvanic corrosion.

Both glass and plastic substrates have been examined. Except for certain mechanical properties relating to the use of glass as a substrate material, it has been found to be very good. However, properly selected plastics appear to be equally as good, if not better. Mylar and unplasticized polyvinylchloride have been found acceptable as plastic substrates. Mylar has been utilized for the visual indicators because the slightly translucent quality of this material proves to be an advantage in that it screens the slight flaws in the deposited metal film and the initial pinholes caused by corrosion. This advantage tends to cause less confusion to the observer during the initial corrosion process. White polyvinylchloride, polymerized diallyl phthalate, and Lucite have also been found to be acceptable as substrates. Utilization of one type of plastic substrate over another depends primarily on physical properties.

Some plastic substrate materials have been found to be unacceptable. Materials which are plasticized or contain volatile components have generally not been satisfactory. In some cases volatile components will contaminate the metal film during the vacuum deposition process. Substrate reaction with the metal film can also occur at high temperatures and either high or low humidities through degradation of the plastic.

With unsensitized metal films substrate cleaning is an important factor. Absorbed gas layers remaining on the substrate affect the metal films' corrosion rate drastically and erratically. This problem has been lucidly described by another investigator.[2,3] Sensitized metal films corrode faster and more uniformly, generally independent of the type of substrate cleaning utilized. Good cleaning procedures are necessary to provide adherent, uniformly deposited metal films. Generally, any number of different chemical cleaning procedure can be used. Good results have been obtained when the substrates were degreased with petroleum ether followed by rinsing with methanol and drying in a desiccated environment. Substrates must be thoroughly dried prior to film deposition. Preheating and ion-bombardment substrate cleaning techniques were found to be unnecessary when chemically cleaned substrates were used with sensitized metal films.

It was considered desirable to develop indicators which would not show corrosion for an initial time period when an environment was corrosive. During manufacture, and prior to use, indicators may be exposed to a corrosive environment. In many packaging and storage applications the desiccant or VCI material will not protect the packaged hardware item until some time has elapsed. Generally speaking, it is believed that about 48 hours are needed at 60 percent relative humidity and 25 C before protection occurs. The time needed for establishing initial protection of package contents is dependent on many factors, including (1) package configuration, (2) initial package atmosphere, (3) location, type and amount of preservative, and (4) the configuration and amount of free space in the package. The time needed to afford protection to an item immediately after packaging is called the "pull-down" period. "Pull-down" time apparently has been only superficially investigated.[13]

The "pull-down" time has been considered in designing the indicators. The process of corrosion could not be stopped but certain design characteristics could be built into the indicator to prevent the corrosion which did occur from being observed. One technique employed was to use translucent Mylar rather than a transparent substrate. Another technique used was to reduce the salt concentration to chromate inhibitor concentration to a low level (i.e., a 4/1 sensitization). These techniques produced about two day delay periods in which the apparent corrosion process was drastically reduced.

Electrical instrumentation was designed so that a signal would be generated only after the electrical resistance of the indicator had increased by a factor of 100. The desired delay was then built into the system. This design also permitted the use of non-temperature compensated electrical indicators.

Changes in temperature affect the electrical resistance of the metal film. The change in electrical resistance required to signal a corrosive environment is far greater than any change which would be induced by temperature fluctuations in normal packaging and storage applications. The electrical resistance for a standard thin metal film corrosion indicator changes only by a factor of about two, when the temperature increases from 70 F to 170 F.

Another technique was needed to produce delay periods greater than about 48 hours. Increase in the amount of chromate-to-chloride ratio beyond the 4/1 sensitization produced greater scatter and sporadic responses in replicate indicators. It does not seem practical to try to increase indicator delay times beyond that now obtained by using the above described sensitization methods. A desirable indicator should not corrode during manufacture, storage, placement in the package, and package "pull-down" period, but should corrode rapidly thereafter if the package wall is ruptured.

The use of VCI for packaging the indicator apparently gives an additional (beyond 48 hours) delay. Though tests are not complete, they show that keeping the indicator in VCI (in Daubert Chemical Company VCI impregnated Mylar bags) gives temporary delays up to about six days when the indicator is removed from the VCI environment and placed in a mildly corrosive environment. The adsorbed VCI protects the indicator from corrosion as long as the VCI is present. When the indicator is removed from the bag, the very small amount of VCI slowly vaporizes and the indicator corrodes normally with its two day "built-in" delay. The VCI gives only a temporary delay until it vaporizes from the indicator surface and no longer offers corrosion protection. Tests are currently underway to document this delay-inducing technique.

The desirability of having indicator delay periods greater than about 48

TABLE 1—Comparative Corrosion Data on Electrical Cards and Buttons in Various Humid Environments

Degrees Temp. (C)	RH	Time For Resistance > 1000 ohms	
		Electrical Cards*	Electrical Buttons*
25	60	6 days 6 days 5 days	6 days 6 days 8 days
25	80	4½ days 3 days 4 days	4½ days 2 days 2 days
25	95	3 days 3 days 3 days	2½ days Poor sample Poor sample
50	80	12 hrs. 12 hrs. 12 hrs.	8 hrs. 12 hrs. 12 hrs.
50	95	12 hrs. 15 hrs. 15 hrs.	5 hrs. 7 hrs. 12 hrs.

* Indicators had 4/1 sensitization.

TABLE 2—Condition of Visual and Resistance Thin Film Indicators at First Sign of Corrosion on Hardware Items

Relative Humidity Percent	Temperature, Degrees C	First Indication on Hardware		Corresponding Condition of Thin Film Indicators	
		Bearings	Shim Coupons	Strip	Resistance
40	25	None at 30 days	None at 30 days	5%	100 ohms
50	25	7 days	7 days	50%	infinite
60	25	7 days	7 days	50%	infinite
80	25	6 days	6 days	100%	infinite
40	50	6 days	6 days	100%	infinite
50	50	4 days	3 days	100%	infinite
60	50	4 days	3 days	100%	infinite
80	50	1 day	1 day	100%	infinite
95	50	16 hours	1 day	100%	infinite

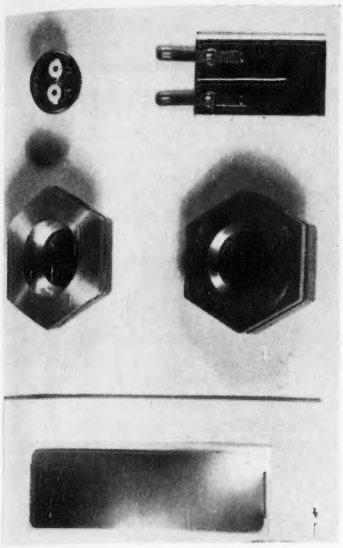

Figure 6—Examples of visual and electrical corrosion indicators and package feed through assembly. Parts are: Top row, left to right, electrical button indicator, electrical card indicator; middle row: package feed through assembly, visual indicator for packaging; bottom: visual card indicator.

hours has not been determined. The technique described above for producing longer, temporary indicator delays might be satisfactory for packaging and storage applications. However, VCI contaminated indicators might furnish misleading information in other applications, such as in laboratory tests. In addition, the small concentration of VCI might affect hardware stored in a small package.

Many tests have been made in which both visual and electrical indicators were exposed simultaneously to the same environment. The rate of corrosion of visual indicators can be varied by changing such factors as the amount and type of sensitization of the metal film. However, upon standardizing these factors the rate of corrosion is reproducibly fixed. Electrical indicators corrode at essentially the same rate; however, the point at which a signal is generated for corrective action by an electrical indicator can be varied. An actual signal can be generated at any point during the corrosion process or traced continuously by an automatic recorder. Electrical indicator probes generally have an initial electrical resistance of about 10 ohms. An increase in resistance by a factor of 100 to about 1,000 ohms is necessary to compare generally with moderately corroded visual corrosion indicators. Presently designed instrumentation for electrical indicators signals for corrective action when the electrical resistance of the metal film reaches 1,000 ohms or higher.

Rectangular electrical card indicators with a large sensitized area (1.75 square inches) have been developed and compared with round electrical buttons with a smaller sensitized area (0.12 square inches). These tests were made to determine the effect of varying the sensor sensitized area (Table 1). Corrosion does not affect the resistance of the thin metal film until at least 30 percent of the area has corroded. At that time the individual corrosion areas (or spots) begin to contact each other, causing sharp increases in resistance with small amounts of additional corrosion. Thus, several small corrosion spots suitably located on a small sensitized area can cause an apparent failure when the environment is not really very corrosive. One corrosion spot on a very small area could trip the alarm needlessly. It is possible to have too small a sensitized area if the corrosion process is not completely uniform.

In addition to the effects of having too small a sensitized area on electrical sensors, the area will also affect the upward "break" in the electrical resistance response curves. This break occurs when corrosion becomes continuous across the thin metal film, thus opening the circuit. If the strip is very narrow, a small amount of corrosion will cause a severence of the film. Thus a signal might be generated even if only 20 percent of the total film area were corroded. On wider strips, 50 to 75 percent corrosion might be needed before bridging occurs. In the sensor sizes selected, the resistance break starts at greater than about 30 percent total corrosion. Obviously, a sensitized area which was too small would perform erratically and would have shorter delays (i.e., less time until the upward "break" occurred).

As mentioned, tests were also made comparing the behavior of visual and electrical indicators with bulk metal hardware. Tests were made in a variety of corrosive environments using polished steel shim stock coupons and ball bearings. Table 2 summarizes some of the test data showing the condition of the visual and electrical corrosion indicators at the point where the first indication of corrosion was observed on the hardware items. No quantitative measurements of corrosion on the hardware were undertaken.

Indicator Designs

Many different configurations of visual and electrical indicators have been evaluated during the course of this program. Many configurations were discarded because of complexity, cost, minimum foreseeable usefulness, production difficulties, and handling problems. A number of different prototypes of both visual and electrical corrosion indicators are being produced for field service testing. (See Figure 6.) These will be described in the following paragraphs to acquaint the reader with what a useful visual or electrical thin metal film corrosion indicator looks like.

Each type of visual and electrical corrosion sensor gives a rapid, sensitive, and reliable indication of the progress of corrosion. The sensors qualitatively provide a cumulative history of corrosive conditions within the environment. Corrosion is indicated visually by a progressive color change from silver-grey to orange or rust-red. The indicators are packaged in desiccated cans to prevent their being corroded prior to use.

The original concept of a single "band-aid" type visual indicator evolved into several different configurations which proved most satisfactory for different applications. One visual indicator consists of a 1″ × 3″ × 0.010″ strip of translucent plastic onto which is deposited a sensitized iron surface. The strip is stapled to a 2″ × 4″ card of polyvinylchloride. The sensitized iron surface is downward against the card, which provides three advantages: (a) the sensitized surface cannot be easily contaminated by handling; (b) the film is viewed from the back-side against an opaque surface (viewed in this manner small pinholes and blemishes are not seen, which otherwise would tend to confuse the observer and cause a scattering of readings); and, (c) any slight dulling of the film owing to the sensitizing salt coating is not apparent. The polyvinylchloride backing protects the indicator and prevents the indicator from getting "lost" in the package. Written instructions, such as the appearance of the word "corroded," could be made to appear on the film in rust-red (sensitized surface). Instructions are engraved on the back of the polyvinylchloride card.

Another visual indicator consists of a small plastic sheet which is folded together like a matchbook. The sensitized iron film is placed on the inside surface of the matchbook while a label and instructions are placed on the matchbook cover. This indicator is small, very inexpensive, and should prove useful in a wide range of packaging-storage applications.

Another visual indicator has been developed in which the sensing element can be observed without opening the container. The sensing element is a flat, transparent Lucite disc mounted in a vapor-proof bushing which fits through a hole in the wall of the package. The disc is held in place by gaskets and a screw-in retainer ring. The size and shape of the indicator housing are identical to those presently in use as relative humidity indicators.

Electrical corrosion indicators with associated accessory equipment and instrumentation have been designed and developed in a variety of configurations. One corrosion indicator model utilizes an electrical circuit which affords a temperature compensation feature. Designed for use with the electrical resistance measuring device, the temperature compensated indicator can be used to measure the rate of corrosion in mildly corrosive environments which fluctuate over extreme temperature ranges. The extent to which corrosion has progressed can be obtained in extremely small increments of the order of one billionth of an inch. These data can be plotted against time to provide information on the rate at which the indicator is corroding. Used in this manner, the indicators reflect the effect of any variation of corrosive condition in the environment.

Resistance card indicators have also been developed. The card is rectangular (1.5 inch by 1 inch) and has two silver coated pins spaced to fit into a two-pin hermetic seal feed-thru connector. The metal film is deposited over the entire

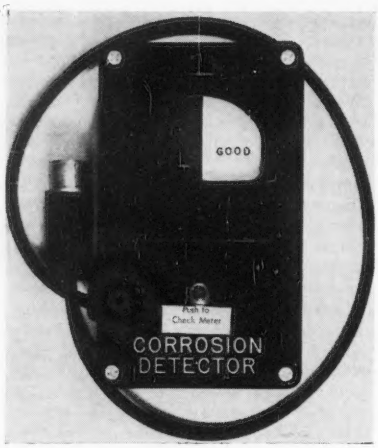

Figure 7—Protype corrosion detection instrument.

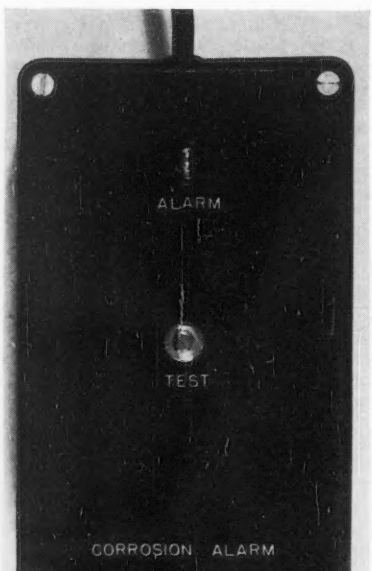

Figure 8—Protype corrosion alarm instrument.

card surface but is sensitized only over the bottom 0.75 inches away from the pins to prevent corrosion at the bi-metallic junction. A line is scribed between the electrical contacts so that the thin metal film forms a "U" shaped pattern. A drop of silver paint is also put over each pin to insure electrical contact.

Two varieties of this type of indicator have been produced which differ in spatial configuration. In one variety the pin-card configuration is such that the card would stick out into the package, and in the other variety, the card would lie flat against the package wall. The large size of these rectangular indicator cards permits a visual observation to be made of the degree of corrosion with considerable accuracy.

An electrical resistance button corrosion indicator has also been produced and will be service tested. The indicator is essentially a round button with a flat face onto which is deposited the sensitized metal film. Two pins extend from the button and fit into the feed-thru connector. The indicator design is essentially the same as the resistance card indicators.

Instrumentation and accessory equipment includes several types of electrical meters, the hermetically sealable package feed-thru connector, and extension cabling. Two types of instruments have been designed specifically for use with the electrical thin metal film corrosion indicators. One meter is a small, battery-operated, portable ohm-meter for simply indicating whether or not the indicator is corroded. A prototype of this meter is shown in Figure 7. The meter contains an integral cable with a connector which will fit either the hermetic feed-thru connector or any electrical indicator directly.

An automatic, battery operated corrosion alarm has also been developed which will rapidly flash a light when the sensor has corroded. (Figure 8.) The light flashes a warning when the resistance of the sensor reaches 1000 ohms (or other pre-selected valve). If the entire circuit, including batteries, is functioning normally, depressing a check button will cause the light to flash. The alarm is designed for installation adjacent to the package being monitored, or remotely if desired. Multiple packages could be monitored by using special station selector switches.

Manufacturing Corrosion Indicators

An integral part of the overall program to develop thin metal film corrosion indicators has been the development of manufacturing techniques and equipment to produce prototype indicators. The manufacturing of each type of indicator has been broken down into its individual steps and analyzed. The critical processing steps for all indicators has been: (a) the cleaning of the substrates; (b) the depositing of the iron film; (c) the sensitizing of the iron film; (d) the completing of the electrical circuitry on resistance indicators; and (e) fabrication of instrumentation. Each of these areas has been extensively investigated and suitable manufacturing jigs and techniques developed to insure obtaining reliable indicator systems.

Packaging and storage of both visual and electrical corrosion indicators has also been studied. The use of desiccated containers has proven to be reliable in preventing premature exposure of the indicators to a corrosive environment. The use of a highly sensitized visual indicator has been used to signify when the desiccant needs to be replaced. The more highly sensitized visual indicator corrodes at a slightly faster rate than the packaged indicators. Indicators have also been satisfactorily sealed into individual packages of aluminized foil or Mylar impregnated with a VCI material. As discussed above, the latter packaging material furnishes an additional delay period since the VCI material acts as a corrosion preventative.

Conclusions

Prototype visual and electrical thin metal film corrosion indicators have been under development since 1955 and have been used in a number of laboratory evaluation programs to monitor corrosive environments. Extensive testing has established those variables which must be controlled during the manufacture, packaging, and storage of the corrosion indicators. Suitable instrumentation and accessory equipment has been designed and developed for use with the electrical and visual indicators. Additional applications for visual and electrical thin metal film corrosion indicators including the detection of ozone, water in jet fuels, packaging and storage of hardware, and atmospheric pollution detection are considered practical from initial investigations.

Acknowledgments

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A Chromatographic Method for the

Separation of Saturated Hydrocarbons From Creosote*

By RICHARD W. DRISKO and HARRY HOCHMAN

Abstract

A chromatographic adsorption procedure has been developed which can separate the saturated hydrocarbons from the other constituents of creosote. Davison Grade 922 silica gel is used as the adsorbent, and *n*-hexane is used as the eluent. Saturated hydrocarbons added to wholly aromatic creosote were recovered quantitatively by this procedure. Ultraviolet spectral analyses of the saturated hydrocarbon fractions separated from both creosote and creosote-petrolatum blends proved the absence of aromatic contamination. 2.3.9

Introduction

THE MOST satisfactory preservative system thus far developed for preventing the attack of marine wood-boring organisms on wooden structures exposed to the sea has been pressure treatment with creosote.[1] The protection afforded by this system varies both with the treatment procedure and the type of creosote used. Fractionation of creosote by distillation or chemical extraction has not resulted in a product with significantly greater preservative properties.[2, 3, 4] Since saturated hydrocarbons are known to be relatively innocuous to higher animals and have been reported to be present in creosote, a method was sought for the separation of this fraction from creosote to determine its toxicity to marine boring organisms.

Heiks, Blum, and Burch[5, 6] have used the β,β'-oxydipropionitrile (ODPN)-petroleum ether selective solvent system[7] for the separation of creosote into fractions corresponding to its aromatic and saturated portions. The Sheridan selective solvent[8, 9] system has been used in the separation of petroleum oils from blends with creosote. Several attempts[9, 10, 11, 12, 13] have been made to utilize a sulfonation procedure for the analysis of petroleum oils in blends with creosote. Neither of the above selective solvent systems nor a sulfonation procedure[14] gave a complete separation. In order to obtain the desired separation, a procedure was developed utilizing chomatography on silica gel which Mair and Forziati[15] had shown to preferentially adsorb aromatic hydrocarbons.

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Method Developed

Apparatus

Apparatus used consisted of a chromatographic column, 9.5 mm (⅜-inch) in internal diameter and 61 cm in length. It was enclosed in a water jacket consisting of a piece of glass tubing 35 mm in external diameter and 50 cm in length fitted with two rubber stoppers and glass inlet and outlet tubes.

Reagents

Reagents used were silica gel (Davison Grade 922) and *n*-hexane (Pure Grade 99 mole percent). This material was used directly without redistillation.

Procedure

To 15 ml of *n*-hexane in the chromatographic column, jacketed with flowing tap water, is added a slurry of 17 grams of silica gel in enough *n*-hexane to permit the mixture to flow freely. Upon settling by gravity, a well prepared column of silica gel approximately 33 cm in height is formed.

While the supernatant liquid is draining to the top of the silica gel column, a small beaker and medicine dropper are weighed. A sample of thoroughly mixed creosote weighing approximately 1.3 grams is added to the beaker and weighed to the nearest milligram. When the surface of the supernatant *n*-hexane just reaches the top of the column of adsorbent, the creosote is carefully added with the medicine dropper. The exact weight of the creosote added is then determined by reweighing the beaker and dropper together with the residual creosote now adhering to their surfaces. Such a weighing procedure is necessary because creosote is not completely soluble in *n*-hexane, and so a weighed sample cannot readily be transferred quantitatively to the column by rinsing the beaker with this solvent.

After the creosote is completely adsorbed on the silica gel, the sides of the glass column are immediately washed down with 1 ml of *n*-hexane. Care is taken that the top of the column is never allowed to become dry at any time during the chromatography. After the wash liquid has drained to the top of the adsorbent column, the glass column is filled with *n*-hexane to a height 18 cm above the top of the adsorbent. The solvent level is maintained within 3 cm of this height for the remainder of the chomatography.

The first 15 ml of eluate collected after the addition of the creosote to the column are discarded. The next 30 ml, which contain all the saturated hydrocarbons, are collected in a graduate, quantitatively transferred to a previously weighed Erlenmeyer flask, and evaporated on a steam bath under a stream of nitrogen. The flask is then cooled to room temperature and weighed to the nearest milligram. The flask is heated on the steam bath for an additional ten minutes, cooled, and reweighed. This procedure is repeated until a constant weight (± 1 milligram) is obtained.

In a blank run, in which no creosote was added to the column of silica gel, 30 ml of eluate were collected and evaporated in the usual manner. One milligram of higher boiling residue was obtained. It is, therefore, advisable that each new lot of *n*-hexane be checked to determine the magnitude of the correction factor.

Experimental Results and Discussion

In establishing the experimental procedure, the separation of the saturated hydrocarbons was followed by observing the change in refractive index of the eluate. Fractions, 1.8 ml in volume, were collected at a flow rate of 2.2 ml/min using an automatic fraction collector. *n*-Pentane was first employed as the eluent and gave good separations when samples were collected manually. However, when used with the automatic fraction collector, the *n*-pentane evaporated so rapidly from the tip of the column that considerable cooling occurred. This eventually resulted in plugging of the column outlet with ice. *n*-Pentane had the additional disadvantage of forming vapor pockets in the adsorbent unless the

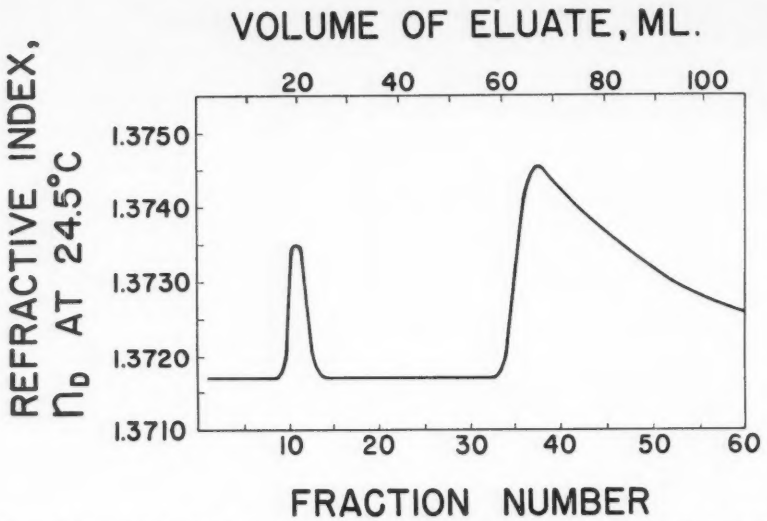

Figure 1—Separation of *n*-hexadecane from a mixture of 0.060 grams of *n*-hexadecane and 1.344 grams of creosote.

TABLE 1—Analysis of Creosote-Petrolatum Blends (Percent Petrolatum by Volume)

MIXED	Found by Chromatography	Percent Error	Found by Sheridan System	Percent Error
2.0	2.0	0	0*	.....
5.0	5.0	0	6	+20
10.0	9.9	—1	11	+10
20.0	19.8	—1	23	+15
40.0	39.7	—0.75	47	+17.5
50.0	49.6	—0.8	57	+14
60.0	59.9	—0.2	64	+ 7

* Two separate analyses showed no phase separation.

column were jacketed with circulating water to maintain a constant temperature. *n*-Hexane had a sufficiently lower vapor pressure to overcome both of these difficulties; however, use of a water-jacketed column was continued.

All of the preliminary experimentation was conducted using samples of coke oven coal tar creosote from a stock which met Federal Specification TT-W-556a. No saturated hydrocarbons were detected in this creosote either by change in refractive index or by evaporation of that part of the eluate obtained before elution of the aromatic hydrocarbons. When samples of *n*-hexadecane or *n*-octadecane were added to the creosote, the added material was separated quantitatively. The separation of *n*-hexadecane is shown in Figure 1. These two aliphatic compounds were identified by their melting points. Alicyclic *cis*-decahydronaphthalene appeared to be separated completely from a mixture with creosote, as determined by change in refractive index of the eluate. The exact weight of the separated material could not be determined, however, as some loss due to vaporization occurred in the heating procedure in which the *n*-hexane was removed.

A series of blends of the coke oven creosote and completely saturated liquid petrolatum was prepared. The liquid petrolatum was separated from these blends by the method described in Procedure to give the recoveries shown in Column 2 of Table 1. The maximum error obtained by this method was 1 percent. It will be noted that for blends of both high and low petrolatum content complete recovery of the petrolatum was achieved in the 30 ml volume collected.

For comparative purposes the above blends also were analyzed for saturated hydrocarbon content using the Sheridan selective solvent[8, 9] (40 percent by volume triethylene glycol and 60 percent by volume furfural). The furfural was redistilled before preparation of this solution. Babcock bottles, 100 percent with 2 percent graduations, were used with 10 ml portions of the blends. Separations obtained are shown in Column 4 of Table 1. The values obtained for percent petrolatum were usually at least 10 percent high. Because it was more convenient to prepare the blends in terms of percent by volume, all figures in Table 1 are expressed in these terms.

In addition to the separation of blends, saturated hydrocarbons were separated from undiluted creosotes produced at lower carbonization temperatures. With one of these creosotes it was necessary to conduct the evaporation of the saturated portion at room temperature under the reduced pressure produced by a water aspirator to prevent the loss of lower boiling saturated hydrocarbons.

Heiks, Blum, and Burch[5] used the ODPN selective solvent system on 20 creosote oils and obtained saturated hydrocarbon* values ranging between 57 and 67 percent. When this solvent system was used to analyze coke oven creosote in the authors' laboratory, the weight of the saturated hydrocarbon fraction was also approximately double that of the aromatic hydrocarbon fraction. Ultraviolet and infrared spectra of the two fractions, however, were almost identical with each other and with the spectra of the original creosote. It was concluded, therefore, that very little, if any, actual separation into saturated and aromatic fractions had occurred.

When creosote blends were analyzed for saturated hydrocarbon content by the sulfonation procedure[14] for the determination of saturated hydrocarbons in petroleum distillates, the lack of a sharp interface separation, especially when the saturate content was very low, resulted in uncertain volume readings and poor reproducibility. Because a high value for saturated hydrocarbon content was always obtained by both this and the Sheridan procedure, contamination of the upper saturated layers in both methods was suspected. The upper layers from several analyses by both systems were carefully removed with a pipette having a fine capillary tip, washed to remove any of the lower solvent that might be present, and dried over sodium sulfate. Ultraviolet absorption spectra of all these fractions had sharp peaks indicating the presence of aromatic constituents of creosote. Ultraviolet absorption spectra of the saturated fractions from the chromatography of both the coke oven creosote-petrolatum blends and the creosotes produced at lower carbonization temperatures were flat with no absorption peaks.

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* ODPN selectively extracts the aromatic constituents of a hydrocarbon mixture.[5] Since the hydrocarbons reported to be present in creosote are either aromatic or saturated, the ODPN-insoluble fraction is assumed to consist entirely of saturated hydrocarbons.

Leaching Action of High Purity Water On Plastics and Other Materials*

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Abstract

Laboratory tests were made to determine the leaching effects of high purity water on plastics and other materials used in building or lining of high purity water storage tanks. Specifically tested were enamels used in painting interiors of condensate storage tanks, materials used for filter tubes, plastic and metal materials of construction, and miscellaneous coating and linings materials. Materials tested include polyethylene, epoxy resins, phenol-formaldehyde resins, cotton, cellulose acetate, glass fiber, mineral rubber with inert filler mixture, asphalt clay-cement mixture, polyvinyl over sprayed zinc and aluminum, sprayed zinc and aluminum, hydraulic cement, commercially pure aluminum, and coal tar enamels. 4.6.5

Introduction

THE DEFINITION of high purity water varies from industry to industry. Water containing 5 ppm or more of dissolved solids is regarded as high purity water for some industrial processes. Other processes demand large quantities of water containing 0.5 ppm or less of dissolved solids. Water quality requirements of a number of specific industries and processes that utilize high purity waters are described by Friend.[1] Included are data pertaining to power plants, research laboratories, the manufacture of television tubes, pharmaceuticals, photographic supplies, pulp and paper specialties, ceramics, and textiles.

Until a few years ago the only means available to industry for essentially complete removal of soluble salts from water was distillation. Well designed evaporators that are properly operated can provide condensate containing 1 to 2 ppm or less of dissolved solids. However in practice the amount of solids dissolved in the distillate from commercial evaporators is dependent upon a number of factors and may range to 20 ppm.

The development of modern ion exchange resins has made it economically feasible in many cases to produce large quantities of high purity water containing as little as 0.05 ppm of dissolved solids on a consistent basis. Demineralizers at the Linden Generating Station of the Public Service Electric and Gas Company of New Jersey were designed to produce 3,200 gpm of 0.5 micromho water containing no more than 0.2 ppm of dissolved solids and 0.02 ppm of silica. They currently yield a 0.3 micromho water that contains 0.001 to 0.002 ppm of silica. Provisions were made in the design of this plant for expanding the capacity to 4,800 gpm.

The effects of improved technology in the field of water demineralization are apparent in many industries. For example, the availability of higher purity water has promoted advances in the field of power production and in various manufacturing processes. The demand by industry for more high purity and even for water of higher purity is a continuing effort. There is, for example, a great need at the moment for economical means for reducing the content of organic matter in certain process waters to lower levels. Nuclear power plants will demand the purest water that can be produced on an industrial scale.

Equally as important to industry as the means for producing high purity water are the means for maintaining the purity throughout storage, handling and process. This is particularly true in such processes as steam-electric power generation wherein the water is recycled time and again through process, with only a small proportion of high purity make-up water being added. Recycling promotes a build-up in the concentrations of contaminants added by the various materials of the system. In some cases conditions are met wherein the contaminants are deposited in such critical areas of the system that they seriously affect performance and result in costly shut-downs and repairs.[2] In one power plant, deposition of such contaminants on boiler feedwater pumps resulted in a 20 percent decrease in pump capacity.[3] In another power plant it appeared that boiler tube failures may have been caused by dissolving of organic matter from the coating on a distilled water storage tank in the feedwater system and subsequent deposition of this matter on the tubes.[4] The maintenance of water quality is just as important in other processes demanding high purity water. In manufacturing applications the quality of the product can be affected by contamination of the water.

Naturally the industries utilizing high purity waters have devoted a lot of attention to the selection of materials coming into contact with these waters. Numerous materials that have been used under various conditions of service are described in the literature.[1, 4-8]

NACE Unit Committee T-3-F was organized in 1954 to accumulate, correlate and disseminate information pertinent to the corrosion resistance of materials in high purity water. Great numbers of experiments in this regard have been conducted. Such work has been spurred on in recent years by the activity in the atomic energy field. Much of the experimentation covering a wide range of materials and conditions is described in the literature.[5-6, 8-23]

Most of the published data concerning the action of high purity water on materials describe corrosion rates and other effects on the material exclusively. It is recognized that such data are of general significance in predicting the effects of the action on water quality. Dlesk,[5] Binger and Marstiller,[6] and Clark and Medin[8] give some quantitative data concerning the effects of a few materials on water quality. Problems arise where more such quantitative data would permit a considerably better evaluation of high purity water system design. Some hitherto unpublished data derived from experimentation conducted by various groups are presented in this paper to illustrate the sort of data for which there is an increasing need as water purity standards become more stringent.

The dictionary definition of leaching is "to part with soluble constituents under the effect of a percolating liquid." The term as used in this paper covers the solution of any material whatsoever from surfaces exposed to the liquid.

Company "A" Test Data(1)

For many years the American Electric Power System followed the practice of painting the interiors of condensate storage tanks with a bituminous enamel, which had been found to give the best protection for service conditions. Examination of the formulation of such products suggested their general unsuitability for storage of demineralized water or high purity condensate because of SiO_2 and organic leaching.

Consequently the investigation here reported centered around the leaching of organics from materials thought to have otherwise suitable properties and practical methods of application. Two major classes of materials were tested, one comprised of natural and synthetic rubbers, and the other consisting largely of thermosetting resins.

Tests were conducted by immersing panels coated with the materials in glass trays containing 200 ml of distilled water,

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(1) Company "A" is American Electric Power System.

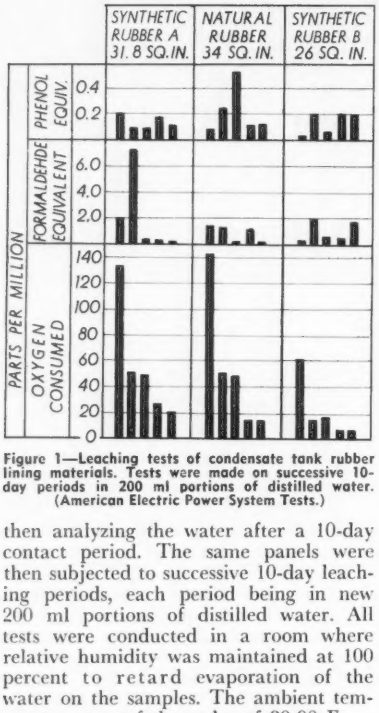

Figure 1—Leaching tests of condensate tank rubber lining materials. Tests were made on successive 10-day periods in 200 ml portions of distilled water. (American Electric Power System Tests.)

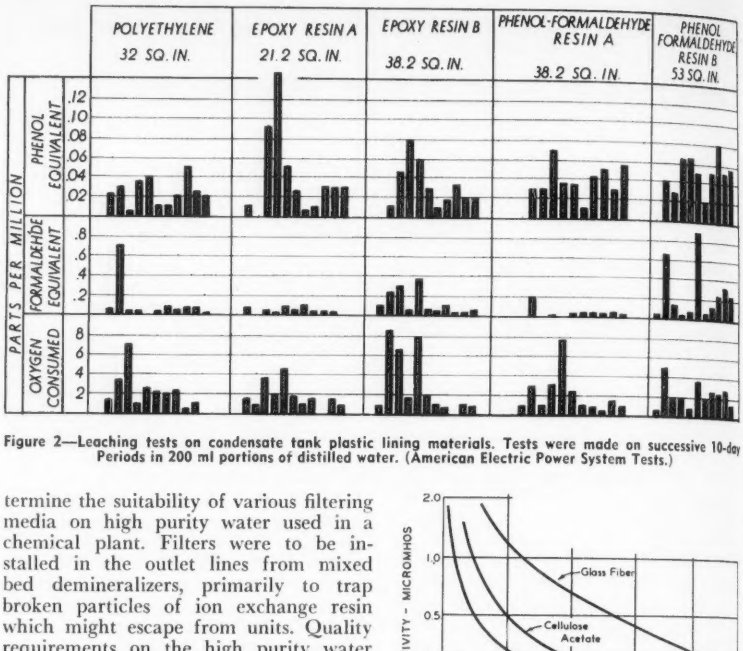

Figure 2—Leaching tests on condensate tank plastic lining materials. Tests were made on successive 10-day Periods in 200 ml portions of distilled water. (American Electric Power System Tests.)

then analyzing the water after a 10-day contact period. The same panels were then subjected to successive 10-day leaching periods, each period being in new 200 ml portions of distilled water. All tests were conducted in a room where relative humidity was maintained at 100 percent to retard evaporation of the water on the samples. The ambient temperature was of the order of 80-90 F.

Since the primary interest centered around a phenolformaldehyde type resin, test procedures for the micro determination of phenol and formaldehyde were developed. The phenol test employed 4-aminoantipyrine in a colorimetric procedure adapted to the Beckman DU spectrophotometer and has a sensitivity of 0.002 ppm. The formaldehyde to determinations also were made by a colorimetric method, employing chromotropic acid. It had a sensitivity of 0.005 ppm. Although these tests are fairly specific, it was found that all of the materials tested gave reactions even though their composition or production did not include phenol or formaldehyde as such. Test results were reported as phenol and formaldehyde equivalents.

As a measure of total organic, determinations were made of oxygen consumed from an oxidizing agent.

The results of the tests on the various materials are shown on Figures 1 and 2. It should be noted that tests on the rubbers shown on Figure 1 were discontinued after 5 cycles, since leaching appeared excessive. The other five materials were tested for 11 cycles and appeared in general to show continuous leaching without significant decrease.

Although none of the test panels indicated rusting for the duration of the tests, the long time effectiveness of such coatings cannot be predicted with any certainty from laboratory tests. The most important factors in lengths of coating life probably are in the application and baking, which are difficult operations in a large field-erected tank.

Company "B" Test Data[2]

Company "A" conducted tests to determine the suitability of various filtering media on high purity water used in a chemical plant. Filters were to be installed in the outlet lines from mixed bed demineralizers, primarily to trap broken particles of ion exchange resin which might escape from units. Quality requirements on the high purity water were that specific conductance should not exceed 0.5 micromho. Water of this nature had been handled successfully at various plants in aluminum, stainless steel, and plastics. Filters previously used had permanent type porous stainless steel media, but plugging problems with some installations indicated operating advantages could be realized by use of a replaceable or "throw-away" type of filter cartridge. The only experience available at that time indicated that filter media made from fibers could be used on waters with conductivity of 5 to 10 micromhos, but that paper media containing asbestos were not suitable.

Tests were conducted on commercially available filter tubes fabricated from cotton, cellulose acetate and glass fiber. The filter tubes were constructed by winding the fiber around a perforated center core to form a cartridge 2½" diameter by 10" long, with a density to remove substantially all particles 10 microns or greater in size. Tests were made in conjunction with operation of a pilot plant demineralizer. As only one conductivity recorder was available, the test set-up had a single tube filter in the demineralizer outlet line, with provision to bypass water around the filter. An in-line conductivity cell was located downstream of the filter and the by-pass.

Each filter medium tested gave significant leaching when first installed; cotton gave the least. Generalized comparative results are shown in Figure 3. It was concluded that any of these filter media could be used successfully but that it would be necessary to flush a filter to waste after installation of new tubes with from 30 to 100 gallons per tube.

An interesting side effect was noted in tests on the glass fiber filter tubes. After the relatively short period of initial leaching, it was found that the conductivity of the high purity water was better after the filter than before the

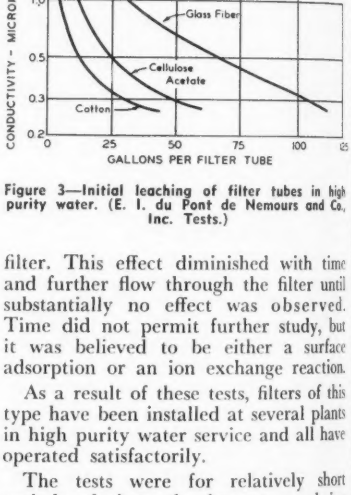

Figure 3—Initial leaching of filter tubes in high purity water. (E. I. du Pont de Nemours and Co. Inc. Tests.)

filter. This effect diminished with time and further flow through the filter until substantially no effect was observed. Time did not permit further study, but it was believed to be either a surface adsorption or an ion exchange reaction.

As a result of these tests, filters of this type have been installed at several plants in high purity water service and all have operated satisfactorily.

The tests were for relatively short periods of time, the longest run being 7,000 gallons per tube. It was believed that the flushing requirements of up to 100 gallons per filter tube represented only a small portion of the effective life of the filter. This has been verified in plant service where the average life is about 150,000 gallons per filter tube. The period of initial flushing to waste is thus less than 1/10 of 1 percent of the useful life of the filter.

Company "C" Test Data[3]

Laboratory scale leaching tests involving several materials of construction were conducted to serve as a guide in the design of demineralized water storage facilities. The following protective coatings were applied to sand-blasted steel panels by a commercial firm specializing in this work, and the panels were submitted to the Company laboratory for testing:

Plasticized polyvinyl chloride
Semi-hard natural rubber
Modified phenolic resin
Polyvinyl top coat over polyvinyl primer

[2] Company "B" is E. I. du Pont de Nemours and Co., Inc.

[3] Company "C" is Public Service Electric and Gas Company.

Polyvinyl top coat over sprayed zinc
Polyvinyl top coat over sprayed aluminum
Sprayed zinc
Sprayed aluminum
Hydraulic cement
Coal-tar pitch base enamel (two types)

Additional test panels were formed from sheets of commercially pure aluminum and a wrought aluminum alloy containing 1.2 percent manganese. The latter panels were welded down their centers.

Duplicate samples of each test specimen were used in the same demineralized water at the ambient temperature, one totally immersed and the other partially submerged. Each specimen was washed in a detergent and rinsed with demineralized water before the test to remove finger marks or other contamination. Test apparatus included polyethylene containers with polyethylene support rods.

The following tests were performed at periodic intervals on the demineralized water in contact wtih the various panels.

Conductivity—All panels.

Iron concentration—All panels except aluminum metal.

Zinc concentration—Sprayed zinc panel.

Aluminum concentration—Panels with aluminum.

Silica concentration—Panels with cement or coal-tar-base coatings.

pH—All samples.

Results of the test are presented in Table 1.

The test results indicate that test panels 3, 4, 5, 6 and 10 (Table 1) did not increase the conductivity of the water significantly. No great amount of base metal contamination was shown present in the water samples in which these panels were immersed. The panels themselves were not affected. It was decided, in addition, that the test results with panels 11, 12 and possibly 13 also were indicative of satisfactory service. Actually, the coal-tar enamel A, represented by panel 12 was selected for certain of the demineralized water storage tanks. Although the leaching tendency of this material on test was slightly higher than that of the first five mentioned above, this particular material was chosen due to its ease of application and because it offered a thicker coating than did the other material.

In tests that preceded those described above, test panels coated with a coal-tar pitch base enamel and others coated with an epoxy resin were installed at various points in a demineralizer pilot plant that was operated continuously. Other panels coated with the same materials were immersed in demineralized water at the ambient temperature in the laboratory. The panels coated with the enamel exhibited no visual changes during a 111-day test period, whereas those coated with the epoxy resin failed completely within 30 days. Conductivity measurements on the water in which the panels were immersed in the laboratory were as follows:

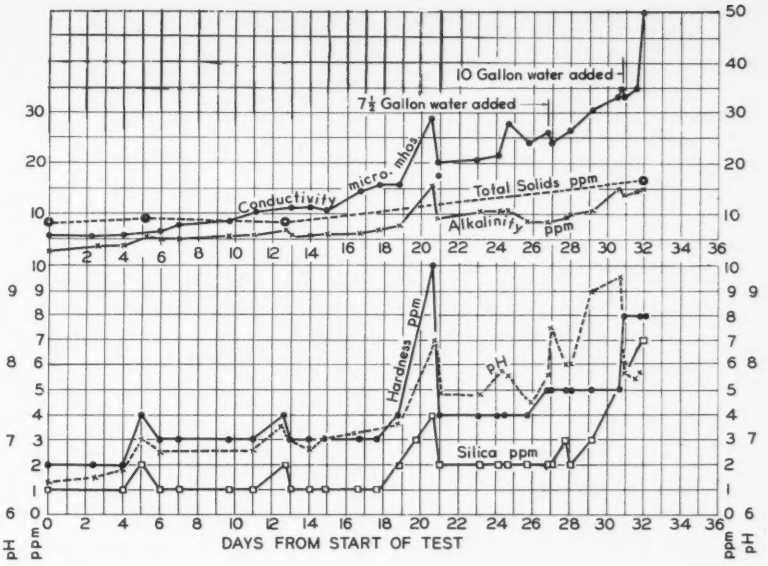

Figure 4—Distilled water storage well lining study (for Product A: mineral rubber-inert filler mixture). (The Tennessee Valley Authority Tests.)

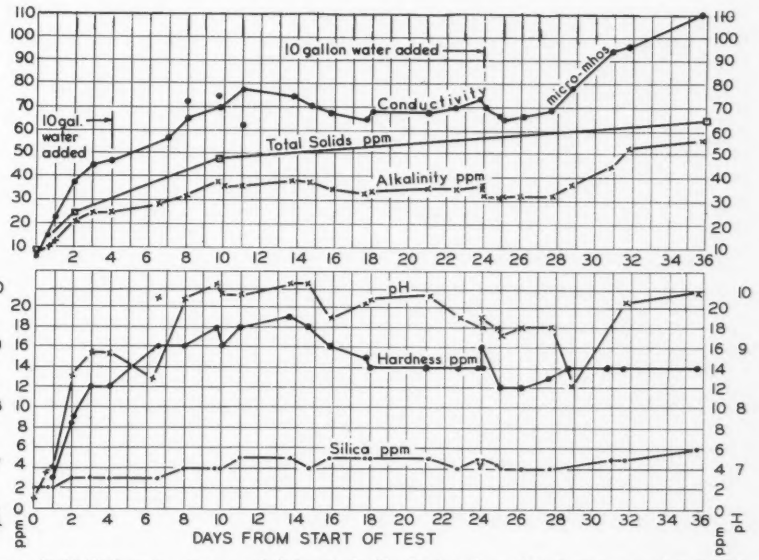

Figure 5—Distilled water storage well lining study (for Product B: asphalt-clay-cement mixture). (The Tennessee Valley Authority Tests.)

Time, days	Conductivity, micromhos	
	Enamel	Epoxy resin
0	0.1	0.1
26	4.2	14
57	5.75	—

In further tests, two tanks about 1 foot in diameter and 2 feet deep were constructed of commercially pure aluminum and wrought aluminum alloy containing 1.2% manganese. These were filled with demineralized water at the ambient temperature. At regular intervals the water stored in the tanks was tested for aluminum content. After about 8 weeks the tanks were emptied, refilled with demineralized water, and the procedure was repeated. The results of the tests are shown in Table 2. These data indicate that some conditioning of the metal occurred during the first tests.

Company "D" Test Data[4]

Test procedures were set up to compare the properties of two commercially available protective coating materials as linings for high purity water storage tanks. There were several reasons for the study, but one of the purposes was to determine the effect of the linings on the quality of stored condensate.

The two materials are identified as "Product A" and "Product B," but it should be noted that "A" is a mixture

(4) Company "D" is the Tennessee Valley Authority.

TABLE 1—Panel Tests*

TEST PANEL	WATER QUALITY							
	Conductivity, micromhos			Analytical Results June 21				
	June 7	June 17	June 21	Fe ppm	Zn ppm	Al ppm	SiO_2 ppm	pH
1. Plasticized PVC	4.73	4.84	6.71	.013	...	.....	....	6.64
2. Semi-hard Natural Rubber	19.00	19.02	19.07	.041	...	.....	....	6.35
3. Modified Phenolic Resin	1.54	1.58	1.78	.009	...	.....	....	6.06
4. Polyvinyl Primer & Top	1.00	1.03	1.15	.010	...	.....	....	6.15
5. Polyvinyl over Sprayed Zn	.69	.86	.93	.012	...	.....	....	6.09
6. Polyvinyl over Sprayed Al	1.11	2.02	2.02	.020	...	.....	....	6.20
7. Sprayed Zinc	25.30	25.30	25.30	.015	395	.....	....	9.41
8. Sprayed Aluminum	15.41	18.39	19.61	.020	...	None	....	8.13
9. Hydraulic Cement	4200.00	4275.00	4700.00	.024	...	.....	8.20	11.80
10. Commercially Pure Al	.88	.87	.91	...	...	None	....	7.20
11. Wrought Al Alloy-1.2% Mn	2.29	2.33	2.44	...	...	None	....	6.90
12. Coal-tar Enamel A	2.17	2.27	2.69	.033	...	.....	.22	6.05
13. Coal-tar Enamel B	2.28	2.86	3.12	.035	...	.....	.02	6.40

* Public Service Electric and Gas Company Test Initiated May 15, 1957; Initial Conductivity 0.77 Micromhos.

Remarks: Panel 1—Exhibited minute blisters by June 17. White substance leaching out.
Panel 7—Lightened in color. Very heavy white deposits. Solution very cloudy.
Panel 8—Darkened slightly in color. Flocculant white substance at interface. Solution cloudy.
Panel 9—Considerable settlement in solution.
Panel 10—Darkened slightly in color. White powdery substance on weld.
Panel 11—Darkened slightly in color. Pitted (etched) on submerged weld. Unless otherwise noted—no color change, blistering, pitting, or rusting of panels.

TABLE 2—Tests on Water Stored in Aluminum Metal Tanks at Ambient Temperatures*

Time, Days	Aluminum Content, PPM	
	Commercially Pure Aluminum Tank	Wrought Al. Alloy (1.2% Mn.) Tank
First Series:		
0	<0.02	<0.02
1	<0.02	<0.02
2	<0.02	0.11
3	0.05	0.10
7	0.08	0.16
14	0.045	0.10
21	0.05	0.10
28	0.035	0.03
56	0.035	0.03
Second Series:**		
7	<0.02	
14	0.05	
21	<0.02	

* Public Service Electric and Gas Company Tests.

** Tanks drained and refilled with fresh demineralized water for the second series of tests.

containing several different compounds, including mineral rubber and an inert filler, whereas "B" is an asphaltic mixture with cement. Two tanks were constructed to simulate conditions in the field, and they were lined according to prescribed procedures. Distilled water was then held in the tanks for given time periods, and the effects of leaching from the linings was determined by analysis of the condensate. The tank with Product A was held at 110 degrees F for 23 days and then the temperature was raised to 178 degrees. The tank with Product B was held at 110 F for 27 days and the temperature then raised to 180 F for about one week.

The results of the tests are shown on Figures 4 and 5. It will be readily apparent that the dissolved constituents as measured by conductivity increased rapidly for Product B and, although more slowly for Product A, still at a steady rate.

Specific tests show much of the pick-up by leaching was accounted for by increased hardness and alkalinity. It is interesting that alkaline materials in Product B should have a very definite effect on the stored water pH, which increased within a week from less than 7.0 to 10.0. Product A, on the other hand, increased the pH of the water steadily throughout the test period. Silica, which could prove difficult to contend with, reached several parts per million for both products. These results confirm, as do those from some of the previous studies, the need for additional work in this field to provide engineers with data that will allow a more comprehensive evaluation of high purity water storage tank designs.

Acknowledgments

The presentation of this paper was planned originally as a symposium to be participated in by several companies. These plans were changed and the authors prepared material provided by the companies for presentation.

The authors gratefully acknowledge the cooperation of the following companies in providing the test data presented in this paper: American Electric Power System, E. I. du Pont de Nemours & Company, Public Service Electric and Gas Company, of New Jersey, and Tennessee Valley Authority.

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Coating Performance in a Marine Environment Evaluated by Statistical Methods*

By FRANK E. COOK and ARTHUR S. MARTHENS

Abstract

Painted steel drums were tested at Kure Beach, North Carolina, and a comparison made of four paints (both for one and two coats) in two colors, on four different steel pretreatments. A method of evaluating the condition of paint is described, and its use illustrated by the tests on the painted steel drums. Statistical analysis of the observed data provides satisfactory assurance of correct interpretation of the results. Film thickness is most important in obtaining durability.

Specification TT-E-485 paint was confirmed as being suitable as a drum enamel. 5.4.5

Introduction

THE NAVY DEPARTMENT, Bureau of Ships had occasion several years ago to investigate the relative durability of protective coatings on 55-gallon steel drums when exposed to a marine atmosphere. An earlier project more limited in scope, showed that a number of factors must be considered. Based on the previous work, it was evident that this would afford an opportunity to make observations which would:

(a) Provide an early measure of deterioration before extensive film failure and rusting.

(b) Be as objective as possible and free from biased personal effects.

(c) Employ numbers on a scale large enough to spread the ratings of 42 different combinations of surface preparation, paint composition, thickness and color.

It is the purpose of this paper to describe the materials and procedures employed and report the results in the numbers of this observation and rating technique, hereinafter referred to as the "numerical" rating together with appropriate statistical analysis and conclusions therefrom. Also included, for comparison, are the ratings assigned by the "overall" observation method.

Experimental Procedure

A total of 138 steel drums were purchased in accordance with Federal Specification RR-D-729a. One hundred and twenty six of these were fabricated by one manufacturer and painted after fabrication. The other 12 drums were fabricated by another manufacturer; the steel for these was pretreated and painted before fabrication. In addition, 12 used drums were cleaned and sandblasted and painted to provide evaluation of such reconditioning. The details of the latter are not included in this paper.

Care was exercised during manufacture of the drums to insure the same cleaning and essentially the same thickness of coating per coat for each paint system. The film thicknesses in Table 1 are the average of three replicate drums except as otherwise stated. Under the heading Drum Designation, the first letter represents the pretreatment (A, B, C or D). The first numeral designates the paint composition (1, 2, 3 or 4), the second numeral the number of coats (1 or 2), and the last letter the color (— olive drab (O) or black (B). Details of pretreatments and paints are given in Table 2.

All drums were laid on wooden racks about 80 feet from mean high tide, as shown on Figure 1. One rack was directly on the sand; the other about 3 feet above the sand. All drums were empty. The original plan contemplated some drums empty, some filled with gasoline or oil, some filled with water, and some filled with inert solid material. However, prior data accumulated indicated that the most severe condition was with the drums empty. One head of each drum faced the sea (east). The drums were set in clusters of three, one drum resting on the other two. Each drum was kept in the same position throughout the test, and kept the same orientation of surfaces toward earth and sky.

Cook Marthens

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Rating of Panels

All observations were made by the same individuals (trained laboratory personnel) although this was not considered essential for the numerical type of ratings.

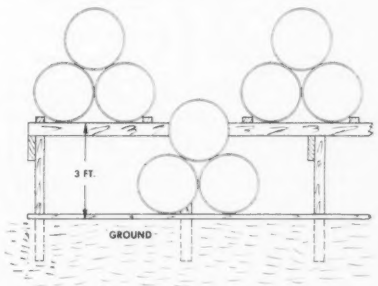

Figure 1—Drums as placed on exposure at Kure Beach, N. C.

Over-all Rating

The laboratory assigned a rating of zero to a newly painted or perfect surface, and 10 to a surface showing complete failure of the protective coating. Values between 0 and 10 thus represent some degree of failure.* In this system, the amount of corrosion, blistering, cracking or other evidence of failure was evaluated visually and the rating assigned through rationalization of the various types of failure.

0 - Excellent (no failure)
1 - 2 Very good
3 - 4 Good
5 - 6 Bad
7 - 8 Very bad
9 - 10 Nearly complete to complete failure

Numerical Rating

The numerical rating system employed templates to cover part of each observed surface, and to concentrate the observer's viewing on one small area at a time, as shown in Figure 2. The template for the head of the drums had 50 openings or windows of essentially equal size, so proportioned that the total surface which could be viewed was half of the drum head. The template for the cylindrical or side surface covered one half of the drum and had 100 windows of equal size, again proportioned so that half of the specimen surface could be viewed. Key marks were

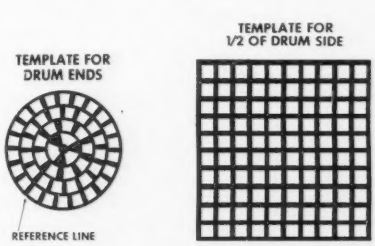

Figure 2—Templates for drums.

* While the direction of the values may not be that usually encountered, it was especially fortuitous in this instance since it enabled simple and direct comparisons between the overall and numerical ratings.

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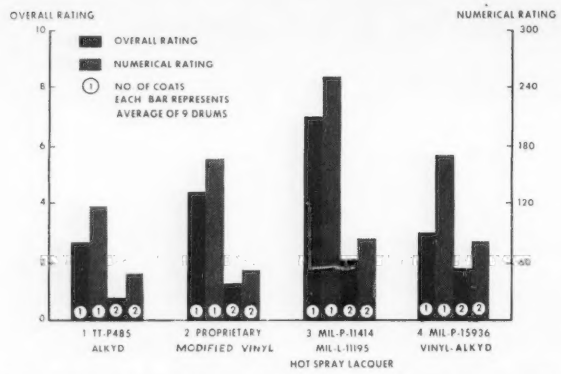

Figure 3—Rating of olive drab coatings after 1 year exposure. Average taken over pretreatments A, B and C.

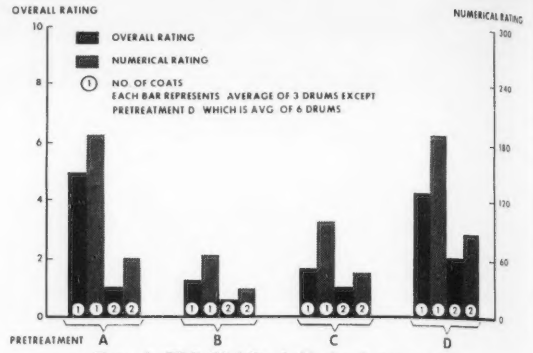
OVERALL RATING
NUMERICAL RATING
OVERALL RATING
NUMERICAL RATING
NO. OF COATS
EACH BAR REPRESENTS AVERAGE OF 3 DRUMS EXCEPT
PRETREATMENT D WHICH IS AVG OF 6 DRUMS
PRETREATMENT A B C D

Figure 4—TT-E-485 (olive drab) after 1 year exposure.

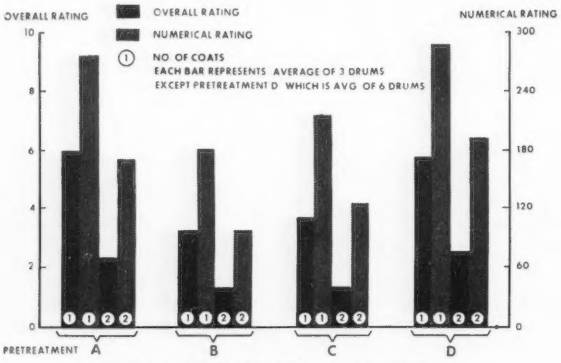
OVERALL RATING
NUMERICAL RATING
OVERALL RATING
NUMERICAL RATING
NO. OF COATS
EACH BAR REPRESENTS AVERAGE OF 3 DRUMS
EXCEPT PRETREATMENT D WHICH IS AVG OF 6 DRUMS
PRETREATMENT A B C D

Figure 5—TT-E-485 (olive drab) after 2 year exposure.

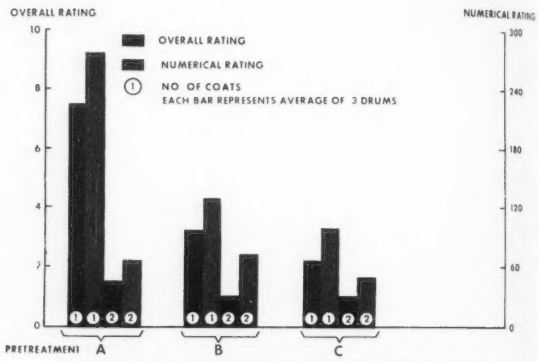

Figure 6—Modified vinyl (olive drab) after 1 year exposure.

TABLE 1—Dry Film Thicknesses

Drum Designation	Mils	Drum Designation	Mils	Drum Designation	Mils*	Drum Designation	Mils
A-11-O	0.8	B-11-O	1.4	C-11-O	1.1	D-11-O	1.2
A-12-O	2.3	B-12-O	3.0	C-12-O	2.9	D-12-O	3.2
A-21-B	1.0	B-21-B	1.1	C-21-B	1.6	Values in this column are the averages of 6 replicate drums.	
A-22-B	2.3	B-22-B	2.4	C-22-B	3.1		
A-21-O	0.6	B-21-O	1.1	C-21-O	1.4		
A-22-O	2.8	B-22-O	2.5	C-22-O	3.0		
A-31-B	1.8	B-31-B	1.9	C-31-B	2.0		
A-32-B	2.4	B-32-B	2.4	C-32-B	2.6		
A-31-O	1.7	B-31-O	1.3	C-31-O	1.6		
A-32-O	2.1	B-32-O	2.5	C-32-O	2.5		
A-41-B	0.6	B-41-B	0.7	C-41-B	1.5		
A-42-B	1.8	B-42-B	2.1	C-42-B	2.5		
A-41-O	0.7	B-41-O	0.7	C-41-O	1.5		
A-42-O	1.8	B-42-O	1.9	C-42-O	2.3		
Average (one coat)	1.01		1.17		1.53		1.2
Average (two coats)	2.25		2.40		2.70		2.3

* Includes pretreatment C; MIL-P-15328, estimated at 0.35 mil thick.
The drums were exposed at the Kure Beach, North Carolina, test site of International Nickel Company (latitude 34° N; longitude 78° W).

employed so that the templates were placed in the same position and the same areas were observed on each visit by the observers.

In rating each drum, the observer merely counted the number of unit areas or openings where any paint failure was visible, without regard to the extent of failure in any area. The number of unit areas which showed any paint failure was counted for each section of the drum separately, (each head, upper half, lower half). Thus the maximum number or score for complete failure over an entire drum would be 300.

The paints and surface pretreatments were as described in Table 2. All coatings were baked.

The paints included in this study were selected because of their good performance and application characteristics demonstrated in previous use and study. For example, TT-E-485 has been standard for the military on drums, pails and military equipment for many years.

The proprietary modified vinyl was the best coating to come out of an industry test series. The hot spray lacquer forms a quick drying and heavy-build system which is finding extensive use. The vinyl-alkyd is used as a top coat on submarine superstructures.

The description of many of the details and variations in this test series are being omitted since they are not essential to a presentation of the results obtained with the two rating systems. It is sufficient to say that all drum cleaning, pretreating and coating operations were witnessed by Navy personnel, the film thicknesses of the paints on the drums were measured, the paints were analyzed for compliance with applicable specifications, and all necessary steps were taken to insure that there was a firm basis for comparison of differences between pretreatments and paints.

Results

The results of the exposure tests in terms of the numerical ratings are provided in Table 3, roughly in order of performance.

Statistical Analysis

The largest component in Table 4 is the effect of two coats of paint against one coat; this factor accounts for more than half of the total variation (i.e. 52 percent.)

The second largest component is the effect of the surface preparations; it accounts for about 17.5 percent.

The third largest component is the contribution of the four paint formulations; this factor amounts to 8.2 percent.

There is some evidence of interaction

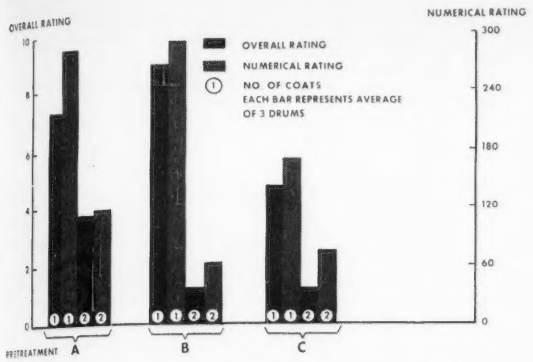

Figure 7—Hot sprayed lacquer (olive drab) after 1 year exposure.

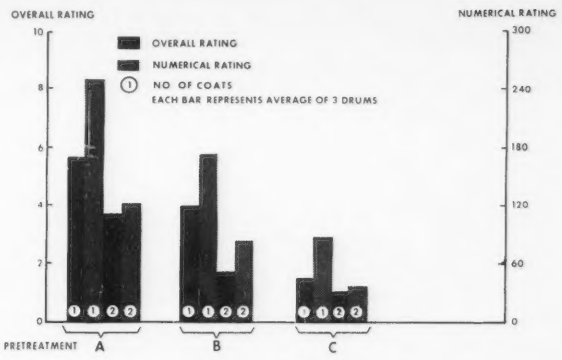

Figure 8—Vinyl-alkali (olive drab) after 1 year exposure.

of the surface preparations with the number of coats of paint.

The other interactions are small; however, an interesting point arises from Table 6 which shows the total scores for the paints 2, 3, and 4 in the two colors. In paint 2, the olive drab pigmentation conferred superior durability. The opposite appears with paint 3 where the black conferred the higher durability. By chance coincidence the difference is about the same, 421 against 419. A statistical test of the four scores shows the difference is highly significant and would not be the result of chance. A conclusion from these data, however, that color *per se* is the controlling factor may not be justified. It has not been established that identical pigments were used to produce olive drab and black in formulation 2, a proprietary paint, that were used in formulation 3 a specification paint. No difference between colors was found for paint 4. Note the improved performance of paint 1 with a value of 1481 as compared to the substantially higher values of the other painted systems.

Discussion of Results on Paints and Pretreatments

On a more specific basis the best result was that obtained with the paint which conformed to Federal Specification TT-E-485 applied over pretreatment B (hot dip phosphate). The vinyl-alkyd coating applied over pretreatment C, wash primer (MIL-C-15328) was a close second; the vinyl alkyd performance over pretreatments A and B however was not equally good. Good results were also obtained with the modified vinyl and the hot sprayed lacquer when applied in 2 coats over pretreatments B and C.

According to the data presented in Table 5, pretreatment C, MIL-C-15328 is, in general, superior to pretreatments A and B, while pretreatment B is superior to A.

The bar graphs, Figures 3 through 8 show the numerical ratings obtained on olive drab paints as compared to the overall laboratory ratings.

Figure 3 shows the overall rating of the olive drab coatings after 1 year exposure with the data averaged for pretreatments A, B, and C. These data are limited to olive drab coatings because it is not possible to obtain a true black color in paint 1, and still retain the compositional requirements. Pretreatment D was also excluded from this comparison because of limited data. Its performance will be shown separately.

TABLE 2—Details of Pretreatments and Paints

Pretreatment	Paint*
A. Acid descaling	1. Enamel, TT-E-485, Type II, Olive Drab for pretreatments A, B and C. Types III and IV for pretreatment D.
B. Phosphate—Same as A followed by JAN-C-490 (150 mg) after fabrication.	2. Proprietary-Modified Vinyl-Olive Drab and Black.
C. Same as A, followed by coating pretreatment (wash primer) MIL-C-15328 after fabrication.	3. Hot spray Lacquer MIL-L-11195 Olive Drab and Black. MIL-P-11414 Primer used in 2 coat system.
D. Phosphate—abrasive cleaned, followed by JAN-C-490 (40 mg) prior to fabrication.	4. Vinyl-Alkyd, MIL-P-15936, color modified to Olive Drab and Black.

* All coatings applied in 1 and 2 coats.

In Figure 3 each bar represents the average of 9 drums. The solid bars represent the over-all subjective type ratings as made by trained laboratory personnel. The crosshatched bars represent the numerical ratings. It will be noted that the numerical system rated the drums more severely than the over-all system in each case, and by about the same amount. Furthermore, the same conclusions would be drawn from both rating systems regarding relative performance. Again it is obvious that the alkyd enamel, TT-E-485 provided the best results on the drums.

Since TT-E-485 enamel looked best, it is of interest to examine the results of this enamel over each pretreatment individually as shown in Figure 4. The best pretreatment for TT-E-485 was B, MIL-C-490, with 150 mg of phosphate coating. Pretreatment D in which the phosphate coating and the enamel were applied to the sheet steel prior to fabrication, appears about equal to pretreatment A for which the steel was acid pickled, fabricated and then painted. Note the vast improvement in performance when two coats of paint are applied instead of one. This improvement is considered highly significant. The sums of the numerical ratings are also shown in Table 6. A factor of roughly 2.5/1 exists between 1 and 2 coats.

TABLE 3—Results of Exposure Tests of Protective Coatings on Steel Drums Where Each Value is the Average of Three Drums

Drum Designation	12 Months	24 Months
B-12-O	30	99
C-12-O	46	126
C-42-O	38	145
C-42-B	45	183
C-22-O	51	130
C-22-B	64	163
A-12-O	63	168
B-11-O	64	186
B-32-O	64	196
A-22-O	65	172
C-32-B	69	167
B-22-O	70	179
C-32-O	76	155
B-32-B	71	200
B-42-B	79	167
B-22-B	80	197
B-42-B	70	206
C-41-O	87	217
A-22-B	91	233
C-11-O	98	218
C-41-B	89	239
A-32-B	104	219
A-42-B	104	218
C-21-O	106	227
A-32-O	115	243
A-42-O	129	200
B-21-O	129	255
C-21-B	143	159
C-31-B	162	264
B-41-O	173	267
C-31-O	174	264
A-11-O	192	278
B-41-B	195	296
A-52-B*	215	284
B-21-B	220	286
A-31-B	226	287
B-31-B	234	284
A-21-B	240	295
A-41-O	252	295
A-41-B	268	298
A-21-O	276	300
A-51-B*	282	298
A-31-O	285	299
B-31-O	292	290

* Reconditioned drums.

Figure 5 indicates the results with TT-E-485 after two years exposure. Here it is obvious that failure is much more pronounced as compared to 1 year, and the overall and numerical systems continue to rate in the same order. A possible objection to the numerical system for drums appears at this point. This is, that as the rating approaches 300, no further differentiation is possible even though complete failure of the paints has not

TABLE 4—Analysis of Variance (1 Year Exposure)

	Degrees of Freedom	Sum of Squares	Mean Square
Paints 1, 2, 3, 4	3	65,515	21,838
Surface Preparations A, B, C	2	139,124	69,562
Coats 1 or 2	1	413,603	413,603
Colors OD or B	1	7,560	7,560
Interaction Paints X Colors	3	2,603	868
Interaction Paints X Coats	3	18,081	6,027
Interaction Colors X Coats	1	1,301	1,301
Interaction Surface Preparation X Paints	6	24,357	4,060
Interaction Surface Preparation X Colors	2	5,153	2,577
Interaction Surface Preparation X Coats	2	37,497	18,748
Higher Order Interactions	17	32,544	1,914
Replications	84	49,514	589
TOTAL	125	796,852	.....

TABLE 5—Comparison of Pretreatments*

	PRETREATMENT A		PRETREATMENT B		PRETREATMENT C	
	Drum Designation	Numerical Rating	Drum Designation	Numerical Rating	Drum Designation	Numerical Rating
TWO COATS	12-O	63	12-O	30	42-O	38
	22-O	65	11-O	64**	42-B	45
			32-O	64	12-O	46
			22-O	70	22-O	51
			42-B	70	22-B	64
			32-B	71	32-B	69
	22-B	91	42-O	79	32-O	76
			22-B	80		
	42-B	104			41-O	87
	32-B	104			41-B	89
	32-O	115			11-O	98
	42-O	129			21-O	106
			21-O	129	21-B	143
			41-O	173	31-B	162
ONE COAT	11-O	192	41-B	195	31-O	174
	31-B	226	21-B	220		
	21-B	240	31-B	234		
	41-O	252				
	41-B	268	31-O	292		
	21-O	276				
	31-O	285				
Total		2399		1782		1248
Average		171		127		89

* Each rating value is average of 3 drums. Drum Ratings after 12 Months Exposure.
** Markedly out of nominal sequence.

TABLE 6—Sum of Numerical Ratings Compared by Color for 1 Year Exposure

	PAINT NUMBER				
Color	1	2	3	4	Totals*
Olive Drab	1481	2092	3017	2251	7360
Black	None	2513	2598	2365	7476
Totals	....	4605	5615	4616	....

Compared by Number of Coats for 1 Year Exposure

	PAINT NUMBER			
No. of Coats	1	2	3	4
1	1062**	3345	4118	3243
2	419**	1260	1497	1373
Totals	....	4605	5615	4616

* Paints 2, 3 and 4 only; Paint 1 not included in totals since there were no drums painted black in Paint 1.
** Olive drab only; no drums painted black in Paint 1.

occurred, as judged by the overall system. The system of counting the number of unit areas showing paint failure proved effective however for the objective of this experiment. The various combinations of paint formulation, surface preparation paint color, and paint thickness were well spread out over the rating scale after 1 year of exposure.

Figure 6 shows the performance of the modified vinyl after 1 year exposure. This coating performed best over pretreatment C, MIL-C-15328.

Figure 7 shows the performance of the hot sprayed lacquer after 1 year exposure. As previously stated, when applied in one coat the lacquer was that conforming to MIL-L-11195. When applied in two coats the first coat was the hot spray primer MIL-P-11414. It is readily seen that use of MIL-L-11195 in one coat should be avoided, but that good results can be obtained with two coat systems applied over pretreatments B or C.

Figure 8 shows the performance of the vinyl-alkyd after 1 year exposure. This coating performed best over pretreatment C.

Conclusions

The technical accomplishments of this program were to demonstrate that paint procured under TT-E-485 is a superior drum coating, to show the value of greater thickness of paint in achieving protection, and to point out the value of a superior pretreatment. The doubling of the thickness by applying two coats of paint instead of one, doubled the performance. Also the performance of two coats after two years exposure was essentially the same as that of one coat exposed one year.

As a rationalization it is obvious that drums procured for stockpiling, or for long term or severe storage conditions where drum integrity is required, should have two coats of a good paint applied to a dry film thickness of 2 to 3 mils over a good pretreatment.

Certain industry representatives indicated at the time of the test that the cost of a second coat of paint on a 55-gallon drum would be in the order of 25 cents per drum, in quantity. If this is a realistic figure the cost is well worthwhile where additional protection for the drum is desired.

As a matter of general interest it is considered that the severity of the exposure conditions to which this series of drums were exposed, is unchallengable. As previously stated, the exposure was at the Kure Beach, North Carolina test site, 80 feet from the ocean. Furthermore, during the two year test period the drums were subjected to three hurricanes, one of which scattered the drums over a distance of one mile. All but one drum was recovered. There was little or no evidence of paint damage by abrasion or impact.

The extrapolation of test results between marine and inland exposures is always uncertain. Although marine exposure is generally more severe, industrial contamination may make inland exposure equally as severe.

Summary

A simple method of rating paint performance has been tried and found effective. Specification TT-E-485 paint has been vindicated as a drum enamel. The value of certain pretreatments has been demonstrated and the improvement in coating performance on drums due to increased film thickness has been related to time.

Acknowledgment

The laboratory work reported herein was planned and conducted by J. F. Gosse, assisted by L. E. Hersh; supervision was by W. W. Cranmer, all at the time with the Industrial Test Laboratory, Philadelphia Naval Shipyard, Philadelphia, Pennsylvania.

The opinions expressed in this article are those of the authors, and do not necessarily reflect the official views of the Navy Department.

Effect of pH in the Range 6.6-14.0+ On the Aerobic Corrosion Fatigue of Steel*

By F. J. RADD, L. H. CROWDER and L. H. WOLFE

Abstract

The aerobic corrosion fatigue performance of a normalized AISI 1036 steel was determined in a 3 percent NaCl brine of various pH levels from 6.6 to the saturated caustic level (14+). A systematic behavior was observed, a behavior which is believed typical for similar steels. These statistical test findings are summarized in fatigue curve form, and in addition, several special corrosion phenomena are recorded. The principal results are explicable in terms of ferrous hydroxide solubility and oxygen concentration cell behavior as the two primary variables. The oxygen concentration cell system is considered as a pH-sensitive discontinuity to the transformed ferrous hydroxide films. A theory is outlined to explain the principal points observed. Finally, control measure possibilities are suggested. 3.4.7

Introduction

THERE ARE numerous operational environments in which steels encounter fatigue stresses in oxygen-containing electrolytes at a pH of 7 or greater. Drill pipe steels and marine service steels are two examples of this. To combat this problem it should first be determined how the oxygen concentration cells operate on a plain carbon steel and in conjunction with variations of stress and pH to cause structural damage.

Experimental

The steel selected for this study was a normalized AISI 1036 stock (carbon-manganese steel: 0.30-0.37 percent C; 1.20-1.50 percent Mn) consisting of two ¾-inch diameter rods, A and B. Measured mechanical properties for these rods are shown in Table 1.

Rod A furnished sufficient specimens for the 6.6 to 13.0 range, while Rod B provided the specimens for the 1N and the saturated caustic solution tests. These rods were considered to be in excellent correspondence so that the data could be considered in total.

The samples were polished longitudinally with aluminum oxide to a metallographic finish. A reduced section R. R. Moore endurance specimen was employed, and the standard R. R. Moore 2,000 rpm testing apparatus was used with the glass capillarity solution contact arrangement for the electrolyte as shown in Figure 1. The steel corrosion fatigue specimen was not touched by the shaped glass capillary tube which was used with a flow rate of about 6 cc per minute. This system was preferred over the drop-by-drop or the wick method, the latter being somewhat a questionable procedure because of the oxygen cells formed at the wick contact line.

The solutions used contained 30 gm/liter of sodium chloride in distilled water with sodium hydroxide being added to produce the desired pH. Consequently, the system studied was an iron-oxygen-water-sodium chloride one, this not considering the iron-manganese carbides and related sulfide or other inclusions or impurities.

Results and Conclusions

The data are summarized in Figure 2 and in Table 2. Figure 3 shows the metallographic facts of the specific steel that was used. Two photomicrographs herein show the caustic etching effect. Figure 4 is a picture of a so-called filiform corrosion product which was found upon one particular specimen.

Figure 5 is a curve drawn from the literature data of the solubility of ferrous hydroxide, the first visible corrosion product in the oxygen concentration cell attack of iron and steel. Figure 6 shows the appearances of two of the fatigue specimens used in this study.

It is evident that while no simple relationship exists in the basic range of aerobic corrosion fatigue life versus pH, there are several major and characteristic features. The first major point is that there exists a lower family of curves in the pH range of 6.6 and including 11.1 wherein the gains from pH control are of little or no avail and wherein there is no endurance limit.

The second major fact is that there does exist a higher family of curves wherein pH control does involve fatigue life improvements, all the members of this family having an endurance limit. Normally, in corrosion fatigue studies, endurance limits are not defined; instead, permissible stress levels are determined for a certain number of cycles, as is necessary with the lower family of pH curves.

The third major feature is that the saturated caustic brine solution gives a special response, a deteriorative etching effect upon the surface. It is of interest to note that this saturated caustic solution response nevertheless involves an endurance limit, despite the caustic etching herein involved.

Theoretical Analysis of the Aerobic Corrosion Fatigue System

Origins of Oxygen Cells

A difference of oxygen access or availability to a given moist iron or steel surface means that the hydrogen films thereupon are not uniform: one surface, the higher oxygen access area, becomes the cathode by virtue of an increased hydrogen-oxygen reaction. From the oxygen concentration cell so formed, emf's as high as 175 mV have been measured, although these were extreme conditions. Each oxygen cell is, in effect, a miniature acid pump.

At the anode of the cell, a strong acid solution is concentrated. This acid dissolves the steel in the anodic area or crevice. It is sometimes a significant point, as when equipment remains stored, that the anolyte acids have a higher density than the parent electrolyte and this acid concentration will cause gravity settling or flow effects in static corrosion cases. At the cathode there is a basic (NaOH) response from the sodium ions there concentrated. If there are ferrous ions in solution in the vicinity, the hydroxyl ions will react to form ferrous hydroxide, which, with dissolved oxygen, is gradually converted to ferric hydroxide and then to rust, $Fe_2O_3 \cdot 1H_2O$.

On a commercial steel surface, there will be oxygen concentration cells formed at structural discontinuities. This will probably be most evident at sulfide locations and various structural rifts. (As a partial proof of this point, note that a hydrogen-treated decarburized steel wire or a pure iron whisker will rust very slowly.) These structural discontinuities will form a given oxygen concentration cell pattern typical of that steel, its surface coatings and its environment. This oxygen cell and special effects analysis is important, because it explains the nature of the electrochemical processes which occur upon the steel fatigue specimen surface and in the fatigue cracks.

Role of Ferrous Hydroxide

The pH value of the dissociation of ferrous hydroxide is about 9.45. With pH values above this, the common ion effect should rapidly produce a decreased ferrous hydroxide solubility.[1] The ferrous hydroxide film is believed to be unstable in reference to the dissolved oxygen and is believed to be converted to a transparent ferric hydroxide film. If the ferric hydroxide film is next converted to a hydrated oxide in place on the surface, then the repair of the final film will still be controlled by the ferrous hydroxide-ferric hydroxide-hydrated ferric oxide transform route. This accounts for the above focus upon ferrous hydroxide.

TABLE 1—Mechanical Properties of Rods

TENSILE STRENGTH, psi		YIELD STRENGTH, psi		ELONGATION (Percent/2 Inches)	
Rod A	Rod B	Rod A	Rod B	Rod A	Rod B
100,202	100,300	67,248	67,900	30.0	32.5
99,333	99,800	69,301	67,700	28.5	31.5
100,872	99,400	68,788	69,200	28.5	31.0
Av. 100,102	99,800	68,446	68,300	29.0	31.7

*Submitted for publication November 30, 1959. A paper presented at a meeting of the South Central Region, National Association of Corrosion Engineers, Denver, Colorado, October 12-15, 1959.

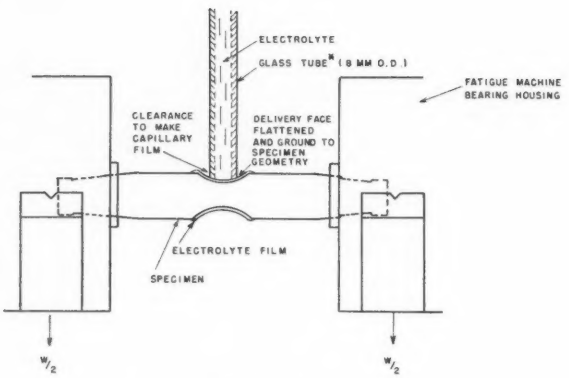

Figure 1—Electrolyte delivery arrangement.

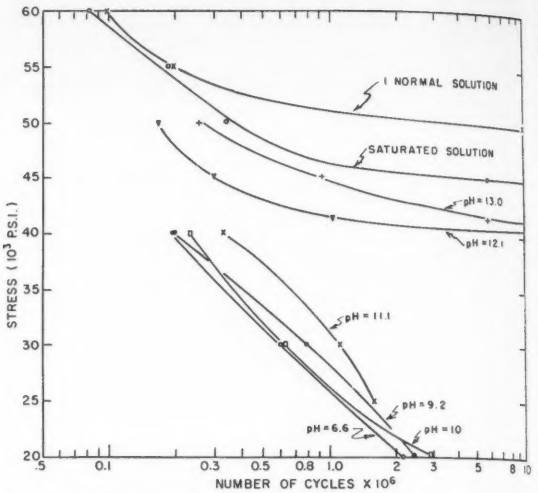

Figure 2—Effect of pH upon aerobic corrosion fatigue life of AISI 1036 steel in 3 percent NaCl solution at room temperature.

Radd

Wolfe

Crowder

FREDERICK J. RADD has been with Continental Oil Company, Ponca City, Oklahoma, since 1952. He did his B.S. Metallurgy Engineer undergraduate work at the School of Mines and Metallurgy of the University of Missouri and his Sc.D graduate work at the Massachusetts Institute of Technology. His research and industrial experience has been with the U. S. Steel Corporation South Works, the G. E. Metallurgy Research Laboratories at Schenectady, MIT, the U. S. Army Ordnance Ballistic Research Laboratory at Aberdeen, and at Boeing Airplane Company in Wichita. He is especially concerned with the low temperature behavior of metals, their oxidation responses, and in their corrosion fatigue properties.

LOUIS WOLFE received his B.S. in mathematics and physics from Northwestern State College of Alva, Oklahoma in 1956. Since his graduation, he has spent two years in the Chemical Corps of the U. S. Army and eleven months doing corrosion fatigue work for Continental Oil Company.

HUGH CROWDER received his B.S. from Oklahoma State University in 1949. He joined Continental Oil Company's research organization in 1954 and since that time has worked on a rather wide variety of problems involving metallography, physical testing and fatigue testing.

Relation of Oxygen Cells to Surface Flms

At zero ferrous hydroxide solubility and the associated film changes, there should be zero corrosion: passivity should prevail. The transformed iron hydroxide film cannot be continuous, however, if the oxygen concentration cell system disrupts it at microanodes which act as tiny acid pumps. Some of these cells are larger and stronger than others so that the film repair capabilities (solution versus replacement rate effects at the cathode-anode interface) of the higher pH levels progressively decrease the oxygen cell population until the last cell is essentially stifled. This oxygen cell smothering is believed due to the transformed iron hydroxide films bridging over the entire cell area. At this point of uniform, continuous film formation, there is an endurance limit in the corrosion fatigue system. A special effect arises, however, when most of the oxygen cells are stifled and only a comparatively few remain.

Cath›de-Anode Area Ratio Effect on Oxygen Cells

In the pH range 10-11.1, the oxygen cell population is sharply decreased. For the survivors the cathode-anode area ratios are extremely large, and the anode currents can be stepped up to 10 or 100 times the previous levels. This is what may cause the abrupt turning down of the pH 10 and the pH 11.1 curves. Furthermore, when the oxygen cell forms at a high cathode-anode area ratio, the further growth and activity of the cell are favored and intensified by the corrosion by-products themselves.

The intensified local pitting produces a most damaging notch effect and thus a lowered fatigue life. The authors believe that this phenomenon explains, assuming that the small experimental effects are real, the curious turning-downs of the intermediate pH curves. As a further curiosity to be found in this pH region, Figure 3 shows a long, hollow filament of oxygen corrosion cell by-products. Such hollow filaments are not infrequently found as by-products of the high cathode-anode area ratio oxygen concentration cell actions as in diethanolamine and similar systems.[2] It is thought that the convection current uplift of the ferrous ions at an anode permits the reaction to form circular, porous, oxygen-reactive iron hydroxide gel structures with the sodium hydroxide present in the cathode area.

Application Guides for The Data

The second point which should be made is that these pH-aerobic corrosion fatigue curves represent systematic, typical data for this particular AISI 1036 steel. While these curves should apply as to general characteristics for similar steels, there is no basis upon which to expect a precise numerical correspondence, this even for other heats of the same steel. The reader is therefore cautioned to use the given data with this view in mind.

Saturated Caustic Solution Effects

The third point is the special effect noted with the rather uniform etching of the steel surface by the saturated caustic—3 percent NaCl system. This seems to be an oxygen cell effect and not a simple flow or solution action. Here it appears that only a limited progressive chemical solution—evidently without crevice accentings—is involved, else there would not be an endurance limit. No hint of a unique embrittlement was observed. The authors wondered if this caustic system oxygen differential cell action with its strong etching effects

TABLE 2—Numerical Values of pH Vs Fatigue Life

pH	Stress Level	Cycles x 10^{-3}	Average
6.6	40,000 psi	201	
"	"	198	195
"	"	187	
"	30,000	536	
"	"	579	601
"	"	690	
"	20,000	2442	
"	"	1613	2196
"	"	2534	
9.2	40,000 psi	188	
"	"	206	199
"	"	204	
"	30,000	750	
"	"	937	790
"	"	684	
"	20,000	2377	
"	"	2363	2449
"	"	2608	
10	40,000 psi	220	
"	"	254	236
"	"	233	
"	30,000	615	
"	"	603	631
"	"	675	
"	20,000	2392	
"	"	3439	2985
"	"	3125	
11.1	40,000 psi	367	
"	"	271	330
"	"	351	
"	30,000	1,056	
"	"	1,168	1,112
"	"	13,114	(Not Used)
"	25,000	1,717	
"	"	1,521	1,566
"	"	1,459	
12.1	50,000 psi	162	
"	"	197	172
"	"	158	
"	"	174	
"	45,000 psi	177	
"	"	216	300
"	"	508	
"	42,500	1,262	
"	"	806	1,034
"	"	237	(Not Used)
"	40,000	9,239	
"	"	22,504	(No Failure)
"	"	21,293	
13	60,000 psi	52	
"	"	58	54
"	"	51	
"	50,000	241	
"	"	242	261
"	"	301	
"	45,000	922	
"	"	888	933
"	"	988	
"	42,500	6,126	4691 (using 3)
"	"	2,302	5885 (using 2)
"	"	5,644	
pH (1 Normal)	60,000 psi	90	
"	"	90	99.7
"	"	119	
"	55,000	247	
"	"	177	194.7
"	"	160	
"	50,000	>20,103	
"	"	1,347	>10,000
"	"	>10,130	
pH (saturated) caustic)	60,000 psi	80	
"	"	78	81.7
"	"	79	
"	55,000	209	
"	"	129	188
"	"	226	
"	50,000	257	
"	"	451	342
"	"	318	
"	45,000	3,646	
"	"	7,886	5,766

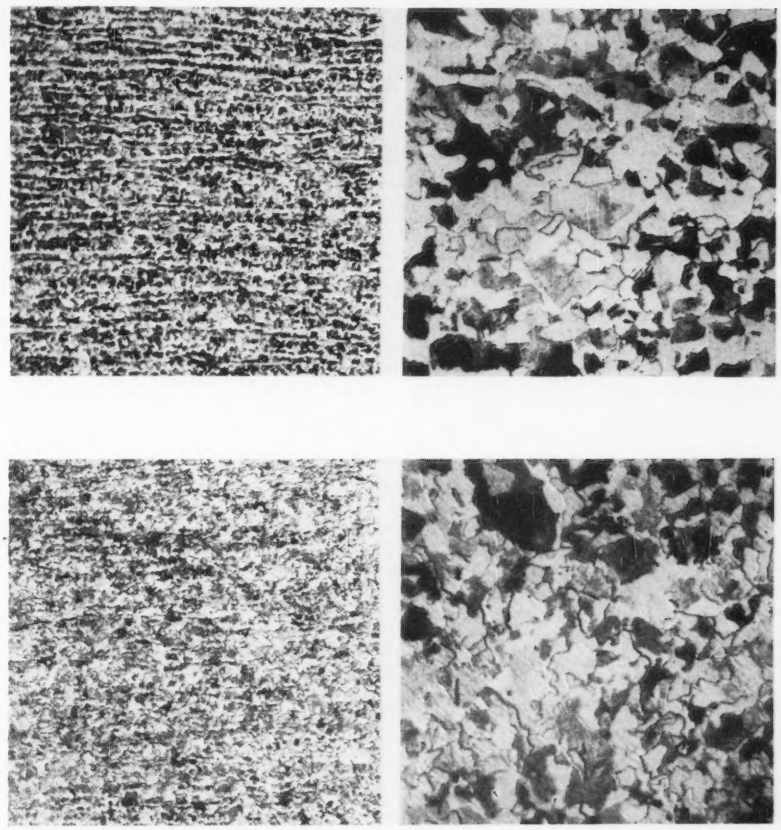

Figure 3—Photomicrographs of AISI 1036 steel specimens. Top—Nital etch views of steel microstructure—normalized 1036 steel. Left, 80X; right, 400X. Bottom—Caustic solution etching of microstructure.

was not the mechanism involved in the cases previously or usually reported as caustic embrittlement.

Fatigue Life Improvement Possibilities and the Physical Basis of the Endurance Limit

The high pH route for corrosion fatigue life improvement is obvious from the data. Not quite as obvious is the engineering feasibility of dissolved oxygen removal from the electrolyte. Sodium sulfite, sulfur dioxide, cathodic hydrogen, hydrogen, and heat can all be used to accomplish this. The prevention or the removal of surface cells (corrosion products, sharp pits, notches, etc.) is one possibility. Another contributory preventative in atmospheric cases is to use a copper content in the steel equal to a minimum ratio of two atoms of copper for every sulfur atom. In some way, therefore, the oxygen cells should be eliminated or minimized.

That pH level at which oxygen cell corrosion is effectively stopped is termed the alpha point. While no attempt was made to determine its exact position in this study, it is known to be $11.1 < \alpha < 12.1$. It is at this alpha point that the aerobic corrosion fatigue curves go from a functionally finite life to a functionally infinite life. If it is desired to extrapolate this atmospheric oxygen data to other concentrations and pressures and other situations, the oxygen corrosion data in a previous paper[3] may be consulted. In brief the relationships $\alpha = \log_{10} \underline{O_2} + K$, where $K = 14.17$ and $\underline{O_2}$ is expressed in grams per 100 ml. solution, serves the oxygen concentration data nicely. In a similar way for a constant pH level, the intensity of the oxygen cell operation in terms of weight loss should be given by $W = K'\underline{O_2} + K'' \log_{10} \underline{O_2}$. The first term is simply due to a given oxygen cell system's operation without any system changes. The second term is the acceleration of cell intensity effect given by the growth *in situ* of the corrosion by-products. If no significant corrosion by-product acceleration of cell intensity is present, then $W = K \underline{O_2}$; then, by analogy, if aerobic corrosion fatigue life is correlated with the dissolved oxygen content at any given stress level, it should be as $\log t = K' - K'' \underline{O_2}$, this for those curves which do not have an endurance limit.

The data appear to show that the endurance limit consists merely of that stress limit at which no surface microcrack growth occurs by mechanical stress action and at which no significant chemical extensions thereof occur (a stable, protective film is present. Here this is a transformed ferrous hydroxide). These

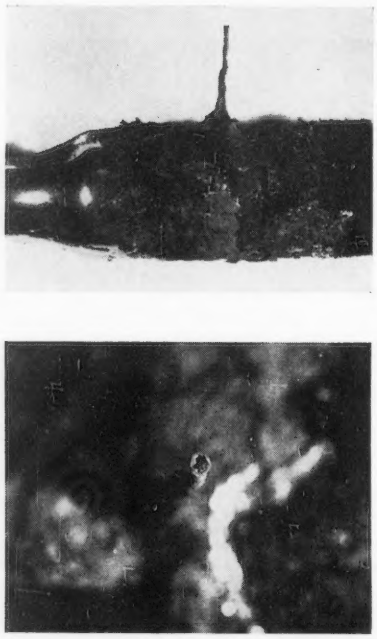

Figure 4—Filiform corrosion product. Top—Hollow filament formed upon an R. R. Moore fatigue specimen rotated at 2000 rpm for 22,504,000 cycles. Specimen was wet with 3 percent brine adjusted to pH 12.1. Approximately 1.5X. Bottom—End view of specimen shown above showing that it is hollow. Approximately 6X.

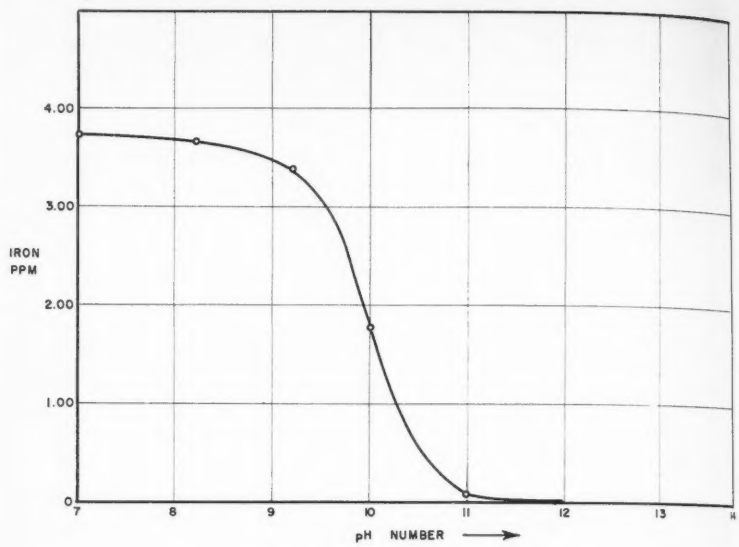

Figure 5—Whitman's solubility of ferrous hydroxide vs pH.

data also imply that the endurance limit can be raised by barrier control or self-repairing film controls.

Finally, some detailed explanation is necessary as to why the endurance limits of the aerobic corrosion fatigue curves of pH levels greater than the alpha point should vary. The most likely explanation is that the greater the film impermeability (of the transformed ferrous hydroxide film) to dissolved oxygen, the less the depolarization of the film-covered cathodes. Hence the analysis suggests that the better and more perfect the oxygen permeability barrier, the higher the endurance limit. This implied conclusion, which represents one of the main results of this work, is in accordance with some of the earlier results of fundamental fatigue studies. These earlier studies showed for a similar steel that vulcanized rubber coatings and Vaseline coatings both had measurable positive barrier effects (giving rightwards and upwards S-N curve displacements) upon the early periods of the fatigue curves.

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DISCUSSION

Questions by A. V. Alessandria, Arabian American Oil Co., New York, N. Y.

1. Why was AISI 1036 carbon steel selected for this test program?
2. What would be the effect on corrosion fatigue of other metallurgical structure conditions as contrasted to the normalized structure employed in the tests? By other metallurgical structures is meant, quenched & tempered martensite, etc.
3. What is the explanation for the decrease in the pearlitic constituent in the basic solution over the unexposed specimen structure?

Replies by F. J. Radd:

1. Carbon-manganese steel AISI 1036 was used simply because it is a fine quality steel which is typical of one sucker rod class.
2. The effect of other metallurgical structures, as the quenched and tempered, is expected to be as per their yield strength and as per their internal friction energy absorption capabilities.
3. There is no real difference, of course. The basic solution apparent difference comes only as an etching irregularity. In the final paper we will have photomicrographs which are nearly identical, that is, between the Nital and the NaOH views.

Comment by F. Lee Current, P. O. Box 117, Oil City, Penn.:

In field usage, we must deal with hot rolled surfaces that are: (1) physically rougher than machined specimens, (2) of lower strength, and (3) of lower carbon content, than is the interior of the article. The difficulty of experimental techniques using hot rolled specimens is extreme. None the less, the above differences are a limitation upon the quantitative extension of laboratory results to field use.

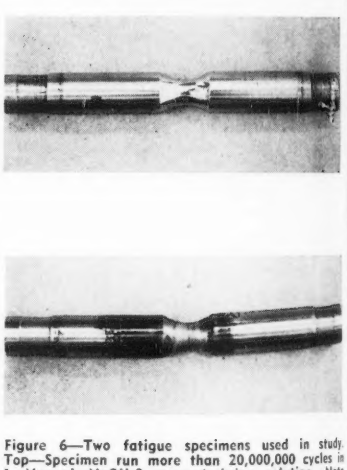

Figure 6—Two fatigue specimens used in study. Top—Specimen run more than 20,000,000 cycles in 1 Normal NaOH-3 percent brine solution. Note bright metallographic finish. Bottom—Fatigue specimen run 7,886,000 cycles in concentrated caustic-3 percent brine to failure. Note etching of surface.

Reply by F. J. Radd:

Your comments are fair and true. Practical steel surfaces are most imperfect. About the only point that I might add is that we have not, as yet, either measured or optimized these steel surfaces. In particular, the as-machined surface and the as-shotcleaned have not been compared in actual field trials of alternating string sucker rod tests. Of necessity, such tests should be field tests. It is my faith that such measures are now fully practical as per the NACE T-IF and the API tests extent. For a summary of such tests the minutes of the API 1959 Corrosion Fatigue Task Force Meeting might be consulted. Finally, I think that the more imperfect surface would have a measurable negative displacement of the corrosion fatigue curve and that such effects are larger than what one might initially think.

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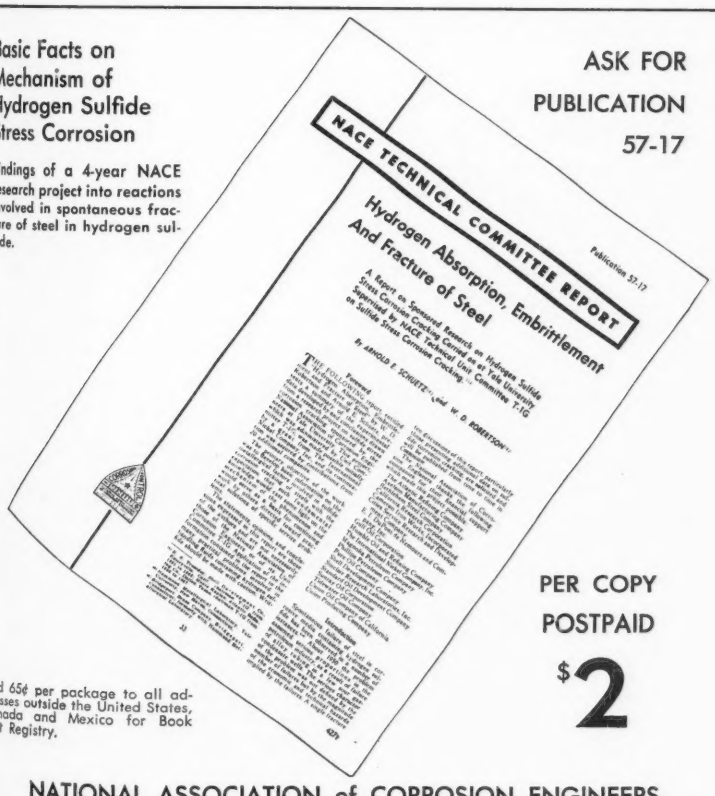

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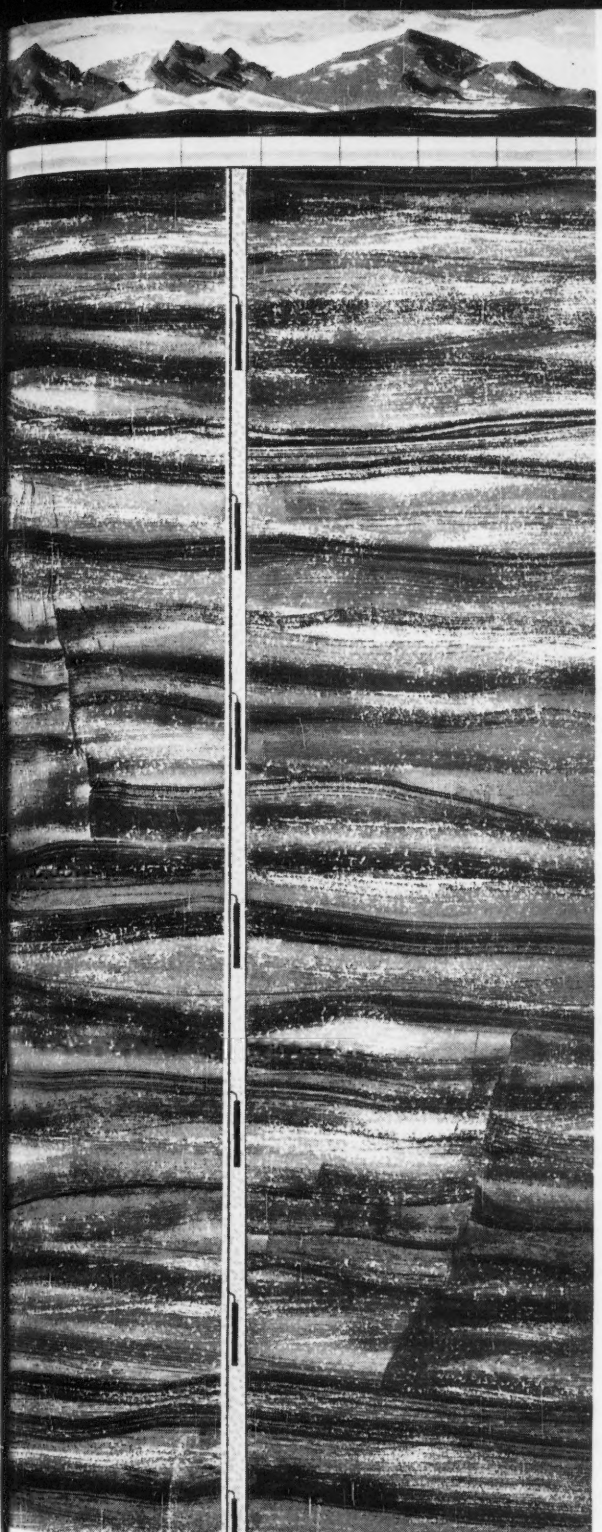

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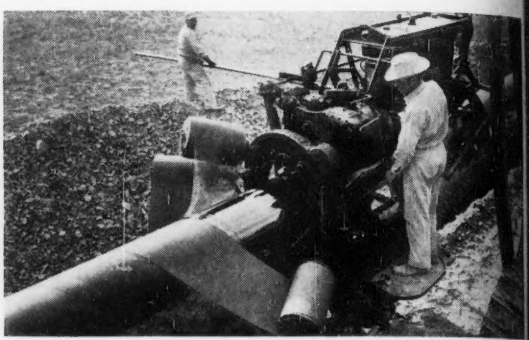
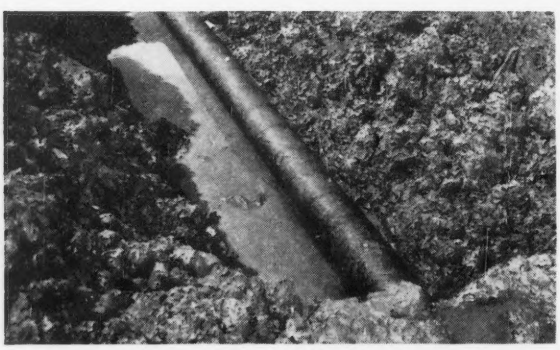
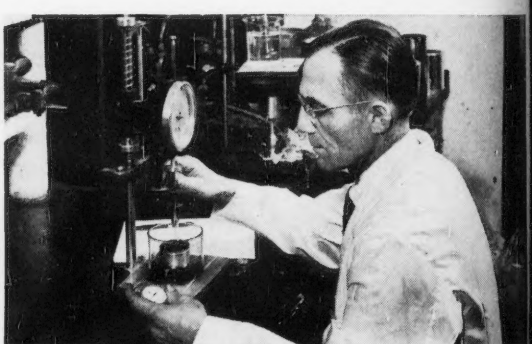
1 *During the next ten years, how much money will be invested in new domestic transmission facilities?*
(a) $3 billion; (b) $6 billion; (c) $16 billion.



2 *The cost of coal tar pipeline enamel amounts to what percentage of a pipeline's total cost?*
(a) 1½%; (b) 2½%; (c) 4%.



3 *This coal tar-protected pipeline, buried in salt water laden soil, was found in perfect condition after:*
(a) 12 years; (b) 17 years; (c) 23 years of service.



4 *What coal tar enamel manufacturer produces every drum of its pipeline enamel to written specifications?*

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